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DEVELOPMENT OF CHROMIUM COMPOSITE ALLOY WITH HIGH TEMPERATURE OXIDATION AND EROSION RESISTANCE

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Directorate of Materials and Processes
Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

Project No. 7381, Task No. 738102

(Prepared under Contract No. AF33(657)-8422 by Metal-Ceramic Engineering Department, Bendix Products Aerospace Division, South Bend 20, Indiana
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NOTICES

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FOREWORD

This report was prepared by Bendix Products Aerospace Division of The Bendix Corporation under USAF Contract No. AF33(657)-8422. This contract was initiated under Project No. 7381, "Materials Application," Task No. 738102, "Materials Pre-Production Processes." The technical work was administered under the direction of the Metals and Ceramics Laboratory, Directorate of Materials and Processes with Mr. Vincent DePierre as the project engineer. This contract was initiated by the Applications Laboratory, Directorate of Materials and Processes.

This report covers work conducted during the period 1 April 1962 to 31 March 1963.

All of the rolling described in this report was performed by the Metalworking Research Division, Battelle Memorial Institute, under subcontract from The Bendix Corporation. Mr. Arnold Gerds, Senior Research Metallurgist, was the Battelle Engineer in charge.

All of the extrusion described in this report was performed at the Experimental Metals Processing Facility, Metals and Ceramics Laboratory, Directorate of Materials and Processes, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

ABSTRACT

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The effects of extrusion and rolling variables on the quality and mechanical behavior of a powder metallurgy chromium-magnesium oxide composite have been studied.

Hot rolling at 2200°F and finish rolling at 900°F with reductions of 40 to 55 percent provided sound, contamination free sheet having a ductile-brittle transition temperature of 45°F in the recrystallized condition. Oxidation, erosion and nitridation behavior were observed to be improved over unalloyed chromium. Preliminary studies have indicated that a strain aging phenomenon may be responsible for the brittle behavior observed with as rolled and stress relieved sheet. Further work is required to resolve this anomaly.

The results of this initial program have indicated that the full potential of chromium composites can be realized with additional development directed toward strengthening, and further retardation of nitrogen diffusion at elevated temperature.

This technical documentary report has been reviewed and is approved.

J. Teres

J. TERES
Technical Director, Applications Laboratory
Directorate of Materials and Processes

(139 pp) (75 fig.) (24 tbls) (2 ref.)

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INTRODUCTION

The adaptation of chromium as a structural material has been retarded in the past due to such problems as low temperature brittleness, embrittlement at high temperature, poor fabricability and inadequate oxidation and erosion resistance. A chromium-magnesium oxide composite, developed by The Bendix Corporation, has shown promise for solving several of these problems. In the extruded condition this composite, designated Chrome-30, combines ductility at room temperature with good oxidation and erosion resistance at temperatures between 2000 and 3000°F. The need for a sheet product possessing these properties prompted the development program described in this report.

The primary objective of this initial program was to develop a Chrome-30 sheet material with a minimum ductile-brittle transition temperature which would be serviceable at elevated temperature. To accomplish this task the following secondary objectives were defined:

1. To establish optimum extrusion procedures for producing sheet bars of uniform quality.
2. To develop sheet rolling procedures for producing sound sheet material.
3. To optimize sheet rolling techniques and low temperature properties.
4. To evaluate the high temperature behavior of optimum sheet.

SUMMARY AND CONCLUSIONS

A total of 55 Chrome-30 sintered billets (93.5 percent chromium, 0.5 percent titanium, 6.0 percent magnesium oxide) were prepared according to procedures developed by The Bendix Corporation. All but three of these billets were found to be suitable for extrusion on the basis of ultrasonic inspection specifications.

Eighteen nickel clad billets were successfully extruded to flat bars at various temperatures and ratios with an average billet-to-extrusion yield of 86.5 percent. The variations in extrusion procedure and subsequent annealing treatment had no significant effect on the hardness, density, microstructure and tensile properties of extruded bars. The average tensile elognation for all extrusions was 23 percent at room temperature and the ductile-brittle tensile transition temperature was found to be 10°F. An extrusion ratio of 10:1 at 2000°F was selected to provide sheet bars for all of the experimental rolling conducted in the program.

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Data obtained from brief forging and wedge rolling investigations provided guide lines for the selection of rolling parameters to be used in preliminary rolling trials.

A brief study was made of sheet bar breakdown rolling at temperatures below the recrystallization temperature. Although initial reductions of 50 percent could be made, rollability was reduced with successive anneals indicating the need for the development of hot rolling breakdown procedures.

An investigation of cladding techniques was undertaken after several unsuccessful hot rolling trials with unprotected and nickel clad sheet bars. A welded frame and cover plate assembly, completely enclosing sheet bars, was found to provide adequate protection for hot rolling.

Breakdown rolling temperatures of 1900°F to 2300°F and reductions of 15 percent to 40 percent were equally effective in producing sheet samples free of external and internal defects. Excessive grain coarsening which occurred at these hot rolling temperatures was undoubtedly responsible for the poor tensile properties obtained from hot rolled sheet. Combined hot and warm rolling, however, refined the grain structure and provided good tensile properties for all hot rolling temperatures investigated.

Maximum room temperature tensile elongation was observed for hot-warm rolled sheets which were fully recrystallized after 40 percent warm work. Tensile elongation ranging from 5 to 12 percent was obtained from specimens with as-rolled surfaces. Specimens which were electropolished provided tensile elongation ranging from 13 to 19 percent. Brittle behavior was observed for as-rolled and stress relieved sheet. Preliminary studies indicate that a strain aging phenomena may be responsible.

Finish rolling of sheet at 900°F to reductions of 40 percent provided sound, contamination free sheets having optimum room temperatures tensile properties. A final annealing temperature of 1800°F provided near equal longitudinal and transverse tensile properties and a ductile-brittle tensile transition of 45°F.

The high temperature properties of optimum Chrome-30 sheet were nearly equivalent to those found previously in the extruded product. Oxidation and erosion behavior were observed to be adequate for many high temperature applications and improvements over pure chromium in nitridation resistance were noted.

Sufficient evidence has been accumulated to show that chromium-magnesium oxide composites not only can be produced in sheet form but possess the potential for solving many of the problems inherent in arc melted chromium. Future areas of study should include:

1. Strengthening mechanisms.

2. Rare earth alloying additions.

3. Welding, joining and forming processes.

4. Scale-up rolling of Chrome-30 and improved chromium composite alloys.

The following tabulations provide a summary of average properties obtained during this program for optimum Chrome-30 extrusions and sheet:

ROOM TEMPERATURE PROPERTIES

	Extruded Bar	Recrystallized Sheet
Ultimate Tensile Strength	48,000 PSI	53,000 PSI
Yield Strength, 0.2 Percent Offset	31,000 PSI	31,600 PSI
Elongation at Room Temperature	23 Percent	19 Percent
Tensile Transition Temperature	10°F	45°F
Impact Transition Temperature	475°F	-
Density	0.237 $lb/in.^3$	-
Hardness	78 R_B	180 Knoop

ELEVATED TEMPERATURE PROPERTIES OF RECRYSTALLIZED SHEET

Test Temperature, °F	Tensile Properties			Oxidation Behavior		
	Ultimate Strength	Yield Strength	Elongation, Percent	Exposure Time, Hr.	Wt. Gain, Mg/cm^2	Nitride Layer, Mils
1800	11,800	9,000	50.0	24	1.3	0
2200	5,000	4,400	28.0	24	4.0	0
2400	3,000	2,000	70.0	24	22.7	4.3

EXPERIMENTAL PROCEDURES AND EQUIPMENT

The extrusion trials conducted for this program were performed at Experimental Metals Processing Facility, Aeronautical Systems Division, Wright-Patterson Air Force Base. All of the sheet rolling trials were performed by the Metal-Working Research Division, Battelle Memorial Institute. Preparation of billets and sheet bars, sheet conditioning and property evaluations of extrusions and rolled sheet were made at the Bendix Metal-Ceramic Engineering Laboratory.

Billet Preparation and Inspection

The three inch diameter Chrome-30 sintered billets used for this program were produced from mechanically blended chromium, titanium and magnesium oxide powders. The nominal weight percent composition of the blended composite was 93.5% electrolytic chromium, 0.5% titanium and 6.0% magnesium oxide. The chromium powder was obtained from Union Carbide Metals Division in four lots which were pre-blended together to eliminate the effects of variable impurity levels on the quality and properties of the Chrome-30 extrusions and sheet. Typical impurity levels of the chromium powder blend were as follows:

Impurity Element	O_2	N_2	H_2	Fe	S	C
Impurity Level, PPM	6600	30	210	590	260	150

A total of 55 billets were prepared in conformance to procedures developed by The Bendix Corporation. Table 17, in the Appendix, provides physical histories for each billet. All billets were machined to the dimensions listed and edges rounded on one end with a 1/2 inch radius to facilitate entry to the extrusion die. After machining, each sintered billet was visually and sonically inspected and coated on all surfaces with a 30 mil layer of flame sprayed nickel deposited by standard wire metallizing techniques. The nickel coating was used to provide lubrication in extrusion and to protect the billet from contamination during pre-extrusion heating. An actual size photograph of Billet 432 after machining and macroetching is shown in Figure 1.

Ultrasonic Examination

Each billet was ultrasonically inspected along the axis of the cylinder and radially over its entire perimeter. Billets were examined by immersion in distilled water with a barium titanate crystal at 2-1/2 mc and quartz at 5 mc. Two 3 inch diameter standard blocks were prepared. The blocks were 2 inches and 4 inches tall. Figure 2, illustration a, shows a reflection from a 0.060 inch diameter flat bottom hole - 1-3/4 inches from the entry surface of the 2 inch high block. The gain has been set to read 1-1/2 inches amplitude using the 5 mc sweep. In Figure 2b, Billet 435 is shown as it appeared during inspection. The trace pictured is from a typically acceptable area in the billet with no defects. The back reflection is slightly diminished. Figure 2c shows an indication in Billet 435 two inches from the top face. Billet 435 was rejected and the indication below the surface was found to be a crack which extended through 25% of the normal area of the billet. Figure 2d shows the trace in Billet 434 in a typically acceptable area. Again,

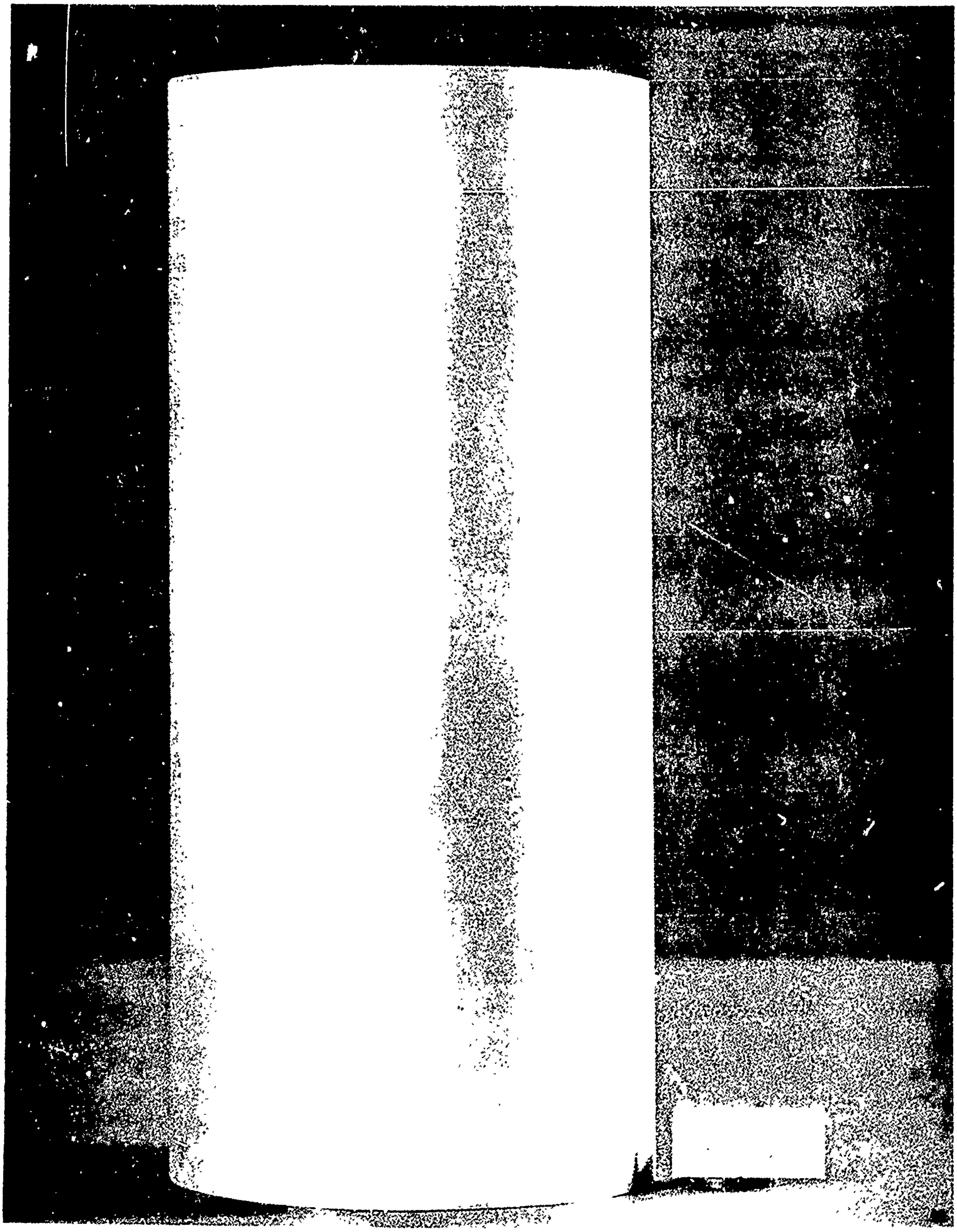


Figure 1 - Macroetched Extrusion Billet #432

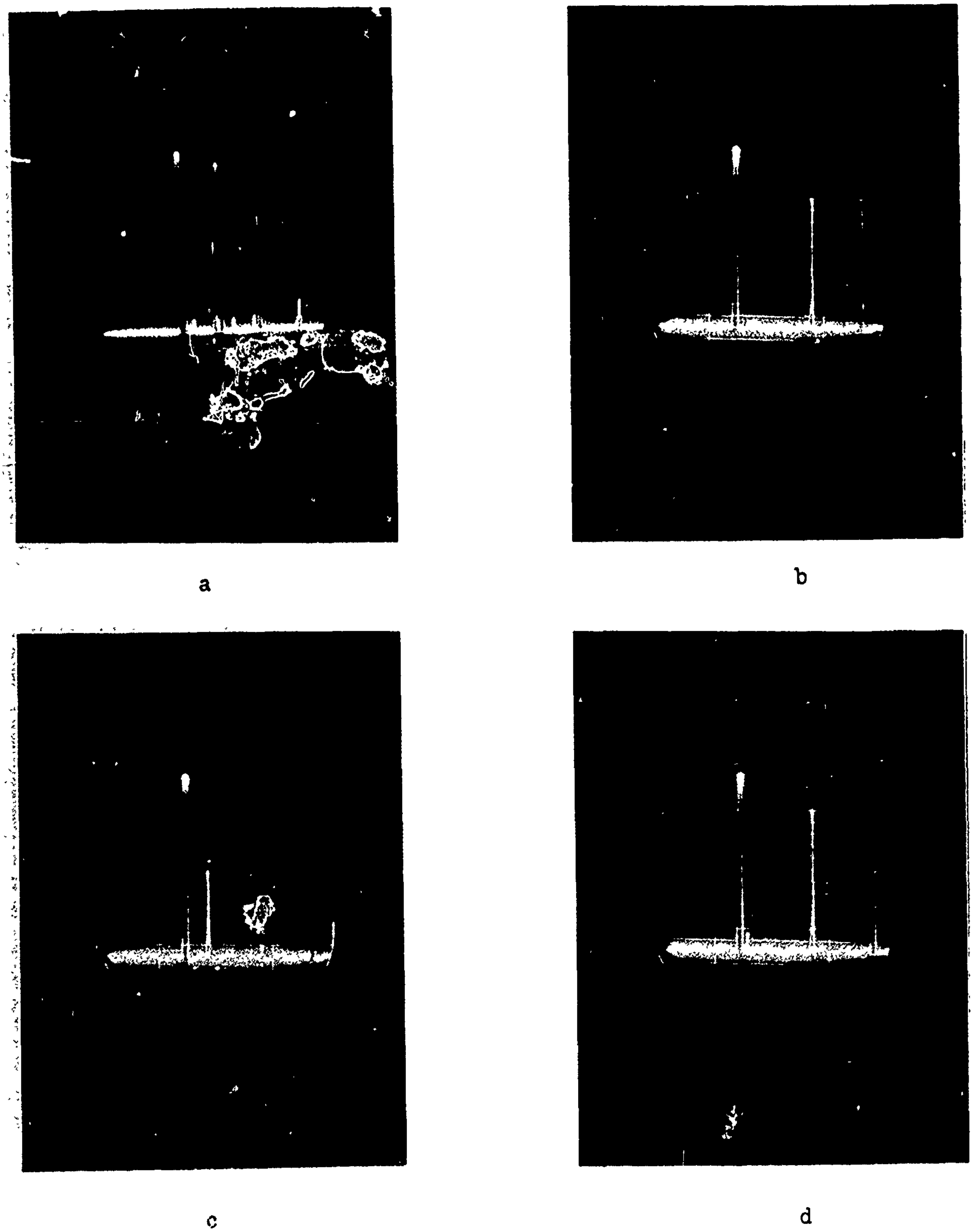


Figure 2 - Ultrasonic Traces of Billets #435 and #434

no defects were indicated but because of the usual porosity in the billet, the back reflection is diminished.

Billet 453 which was rejected because of ultrasonic character is the subject of Figure 3. Figure 3a is the axial view of a defect just below the top surface of the billet and the consequent loss of part of the back reflection. Figure 3b depicts Billet 453 in the radial view with the internal defect about 1/2 inch below the entry surface. Figure 3c is a radial view through the neutral axis bypassing the defect. Figure 3d pictures the radial trace where the entry surface is opposite the defect region showing the signal near the back wall. The defect in Billet 453 and in Billet 452 was a porous area shaped like a half cone that extended half way around the billet with the base of the cone near the base of the billet. The porous area stopped just below the surface of the billet. The axis of the partial cone coincided with the axis of the cylinder. The two billets were rejected and sectioned. The ultrasonic indications were verified by visual examination of the interior of the rejected billets. Of the 55 billets prepared during the program only 3 were found unacceptable by ultrasonic standards.

Microstructure Examination

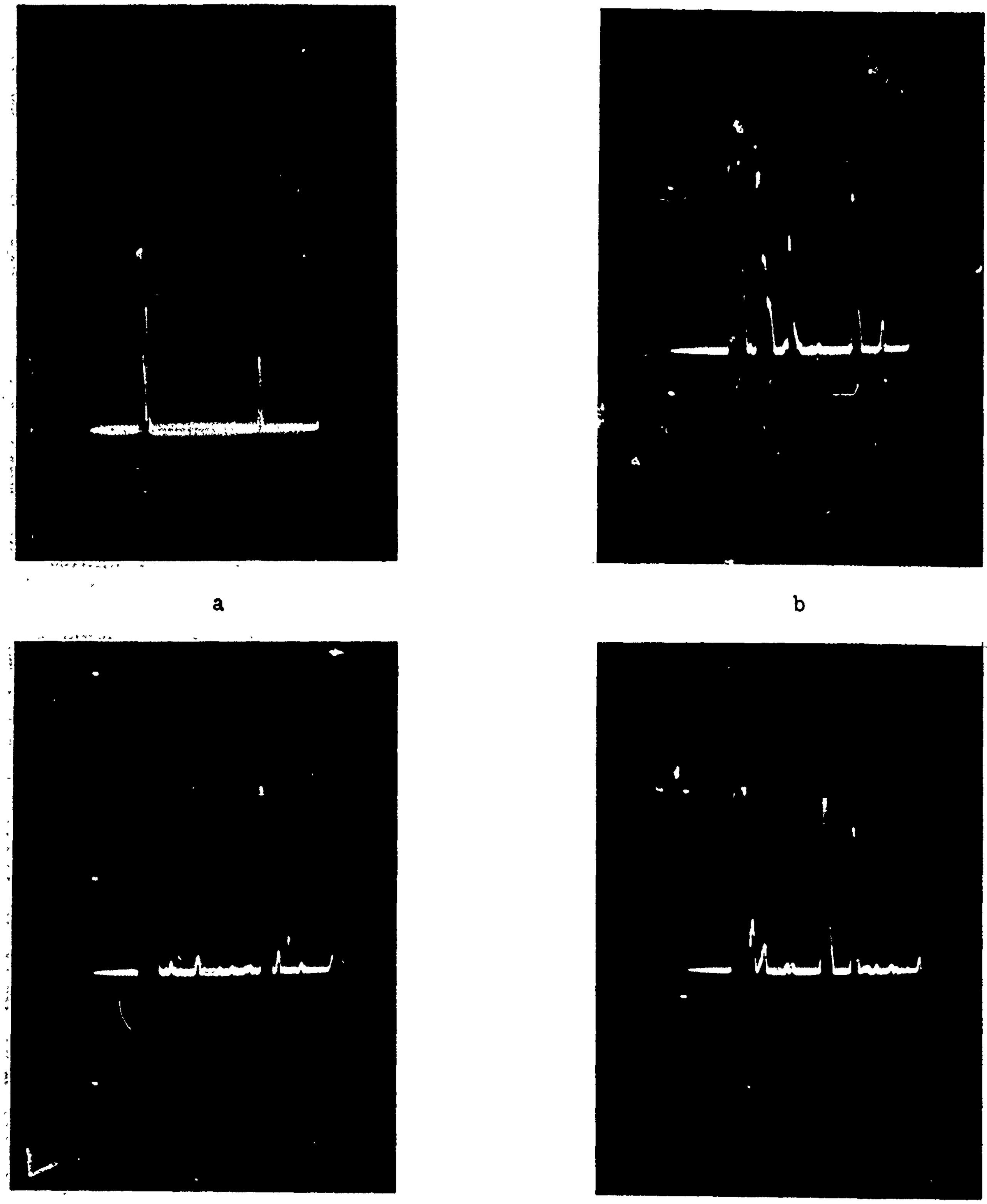
Sections were taken from the ends of each billet to provide comparison of microstructures. The photomicrographs shown in Figure 4 indicate the extremes of oxide particle size and internal pore size observed from examination of specimens from all 55 billets. Photomicrograph a displays a large pore size with relatively large oxide agglomerants while Photomicrograph b shows smaller pores and finer oxide particle size. The microstructure in Photomicrograph c is representative of the majority of specimens examined.

Extrusion and Sheet Bar Preparation

All billets were extruded to rectangular bars using Corning 0010 Borosilicate glass for lubrication. Each billet was induction heated to the desired temperature in an argon atmosphere and the container and die were heated to 800°F prior to each push. Table 18, in the Appendix, lists the extrusion procedure used for each billet and includes a record of extrusion pressures and die condition.

The first 18 extrusions listed in Table 18 were made to study the effects of variable extrusion ratios and temperatures. The results of this study are discussed separately in this report. An additional 34 extrusions were made to provide sheet bars for sheet rolling studies. An extrusion ratio of 10:1 at a temperature of 2000°F was used to provide the necessary rolling stock. Table 19, in the Appendix, provides property data pertinent to these latter extrusions. Extrusions were of sufficient length to permit an average of 5 sheet bars, 5 inches long, to be taken from each. Flattening was required for nearly all sheet bars due to the twisted condition of the extrusions. This was successfully carried out on a hydraulic press by applying moderate pressure to bars heated at 1000°F. The flattened sheet bars were subsequently vacuum annealed at 2000°F for 1/2 hour and examined visually with a 15X binocular microscope to determine the quality of the nickel cladding.

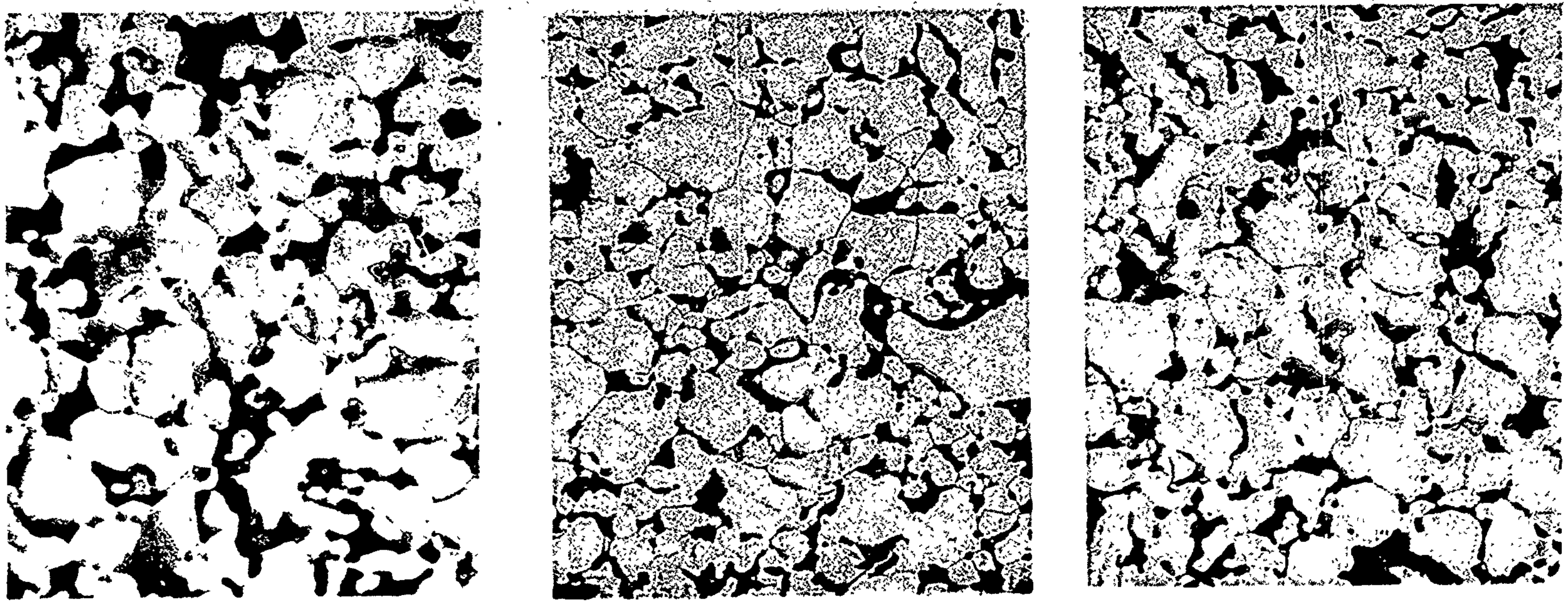
The sheet bars required for framed assemblies were rough sized by milling, finished ground to the dimension shown in Figure 5, and subsequently inspected by wet immersion ultrasonic techniques. A typical framed assembly, shown in Figure 6,



a b

c d

Figure 3 - Ultrasonic Traces of Rejected Billet #453



0-537 200X a | 0-548 200X b | 0-543 200X c

Figure 4 - Sintered Billet Microstructures

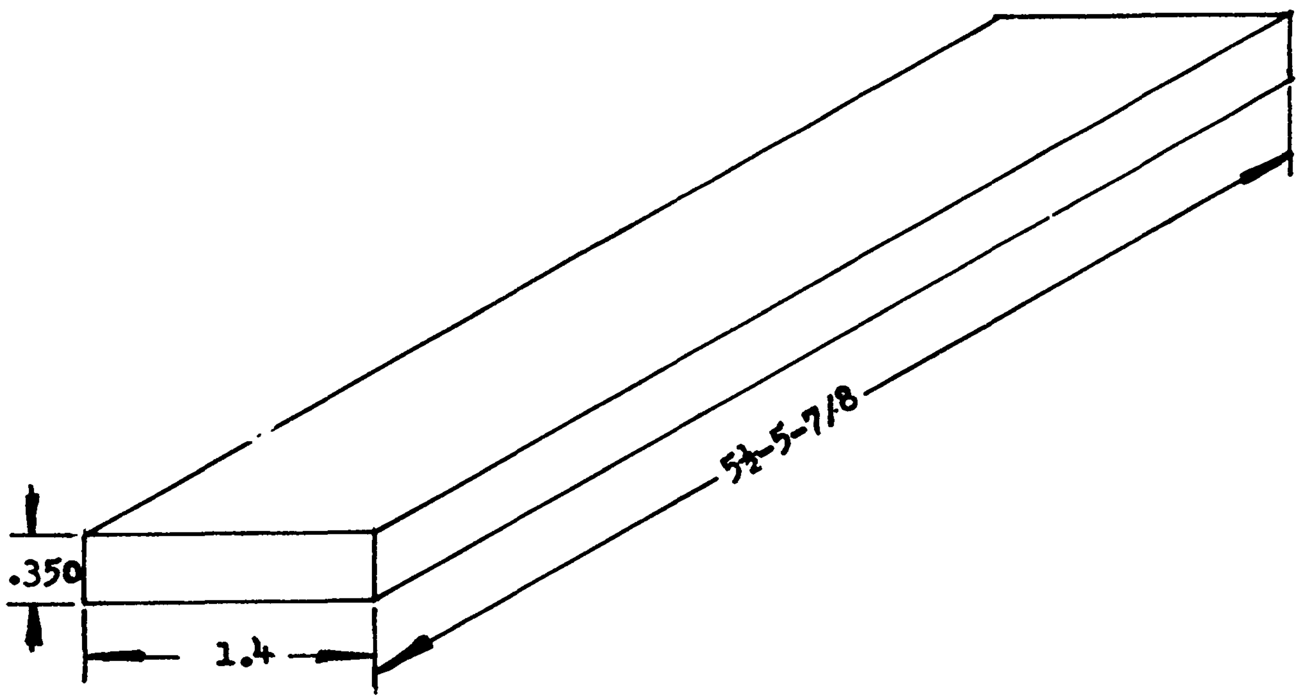


Figure 5 - Sheet Bar Configuration

consisted of two steel cover plates 1/8 inch thick, one rectangular steel frame 0.350 inches thick by 1/2 inch wide on all sides, and a machined sheet bar. The cover plates were attached to the frame by Heliarc welding at the edges and spot welding at intervals along top and bottom surfaces.

Midway through the sheet rolling study, V-shaped markings were observed on nearly all hot rolled sheets after they were removed from the frame. This suggested that the sheet bars may have developed cracks during milling or grinding. Dye penetrant inspection verified the presence of surface cracks and a subsequent investigation isolated their origin to the grinding operation. The sheet bar shown in Figure 7 displays the typical crack pattern revealed by dye penetrant inspection. This condition is believed to have been caused by excessive grinding heat generated by a rapid glazing grinding wheel. At the conclusion of the investigation the following procedure was established and found to be completely satisfactory for the preparation of sheet bars:

Grinding Wheel	- Norton 32A60-H8VBE
Grinding Fluid	- 1 Part Vantrol 700 to 10 Parts Water
Wheel Speed	- 600 SFPM
Table Speed	- 750 IPM
Unit Cross Speed	- 0.050 Inches
Unit Downfeed	- 0.002 Inches per Complete Crossfeed

Rolling and Sheet Conditioning

All of the rolling trials described in this report were performed on a two high laboratory mill (8 inch diameter rolls x 12 inches long) at a speed of 97.8 surface feet per minute. The rolls were operated at room temperature during the warm rolling trials and preheated to approximately 200°F for hot rolling. In all cases sheet bars or previously hot rolled sheet were heated at the specified rolling temperature for 30 minutes prior to the first roll pass and reheated for 10 minutes between each succeeding pass. Sheets rolled at warm rolling temperatures (600-1200°F) were heated in a circulating air furnace. Sheet bars for hot rolling were heated either in an argon or hydrogen muffle furnace or in a hydrizing furnace with an atmosphere consisting of approximately 21% CO, 38% N_2, 40% H_2, 1% CH_4 and no CO_2. The latter was used for framed bars only. Unless otherwise noted all sheet bars were hot rolled or hot and warm rolled in a direction transverse to the direction of extrusion.

Hot rolled sheets were prepared for warm rolling by pickling in hot concentrated hydrochloric acid followed by required annealing, trimming, squaring, and edge sanding. All sheets were examined visually before and after pickling for cracks and other surface or edge defects which might impair cold rolling characteristics.

Tensile Specimen Preparation and Testing

The tensile test specimens used in this program were machined to the dimension shown in Figure 8. The round bar specimen is a modified ASTM bar having a 3/4 inch gage length and a 0.189 inch diameter reduced section. The round bars, used in the evaluation of extruded material, were profiled ground from centers and polished with 4/0 paper to a 10-20 microinch finish for all tests.

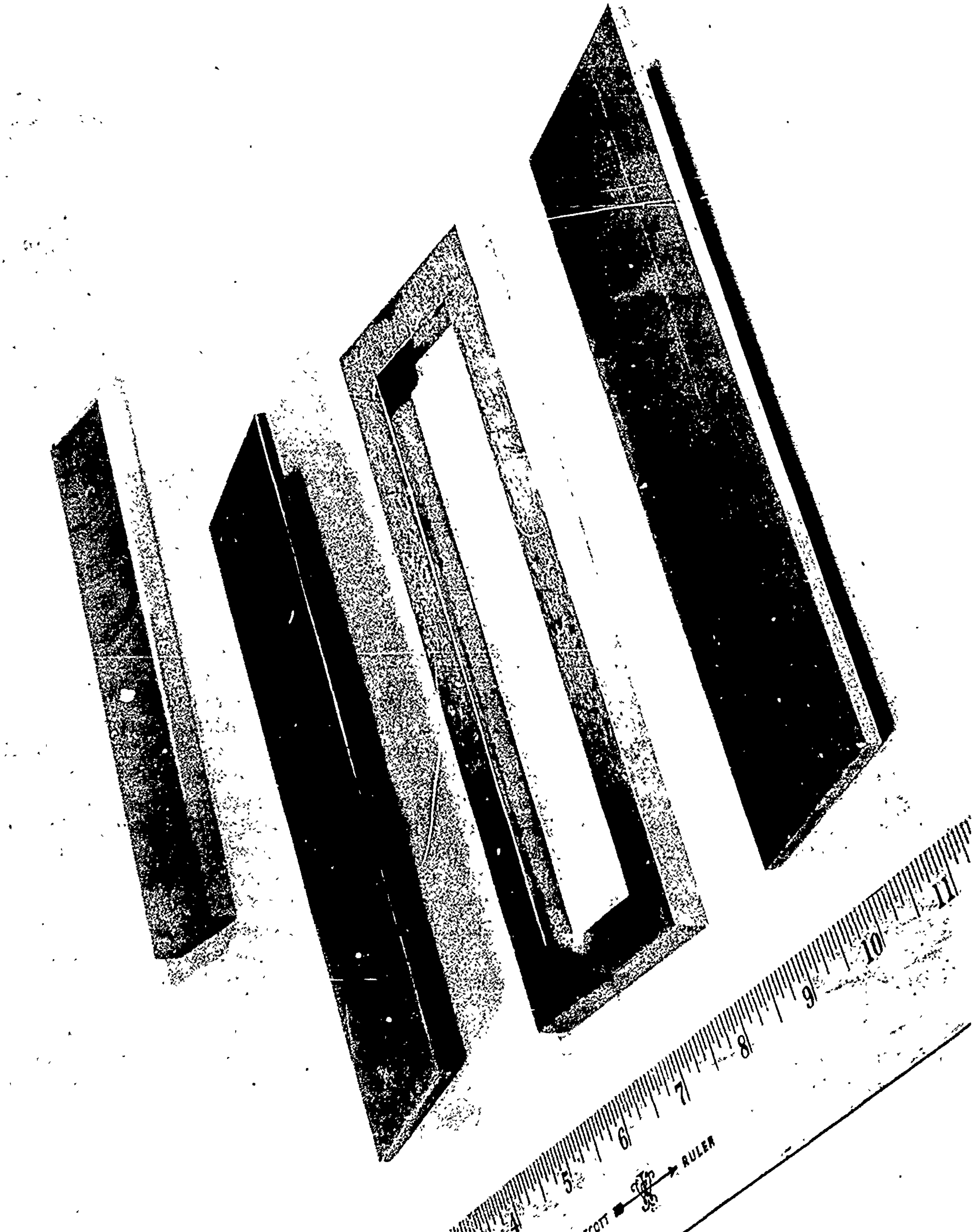


Figure 6 - Sheet Bar and Frame Assembly

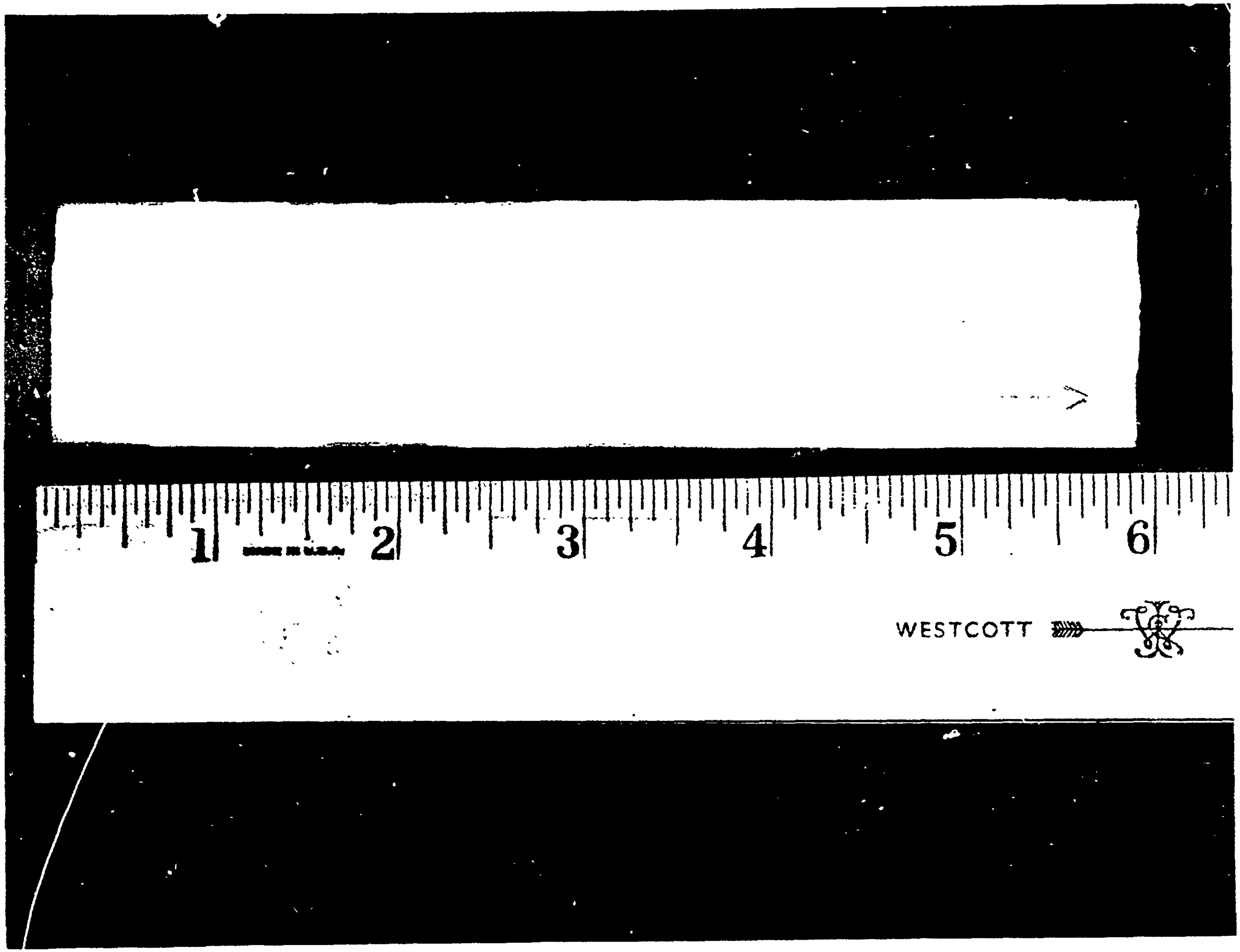
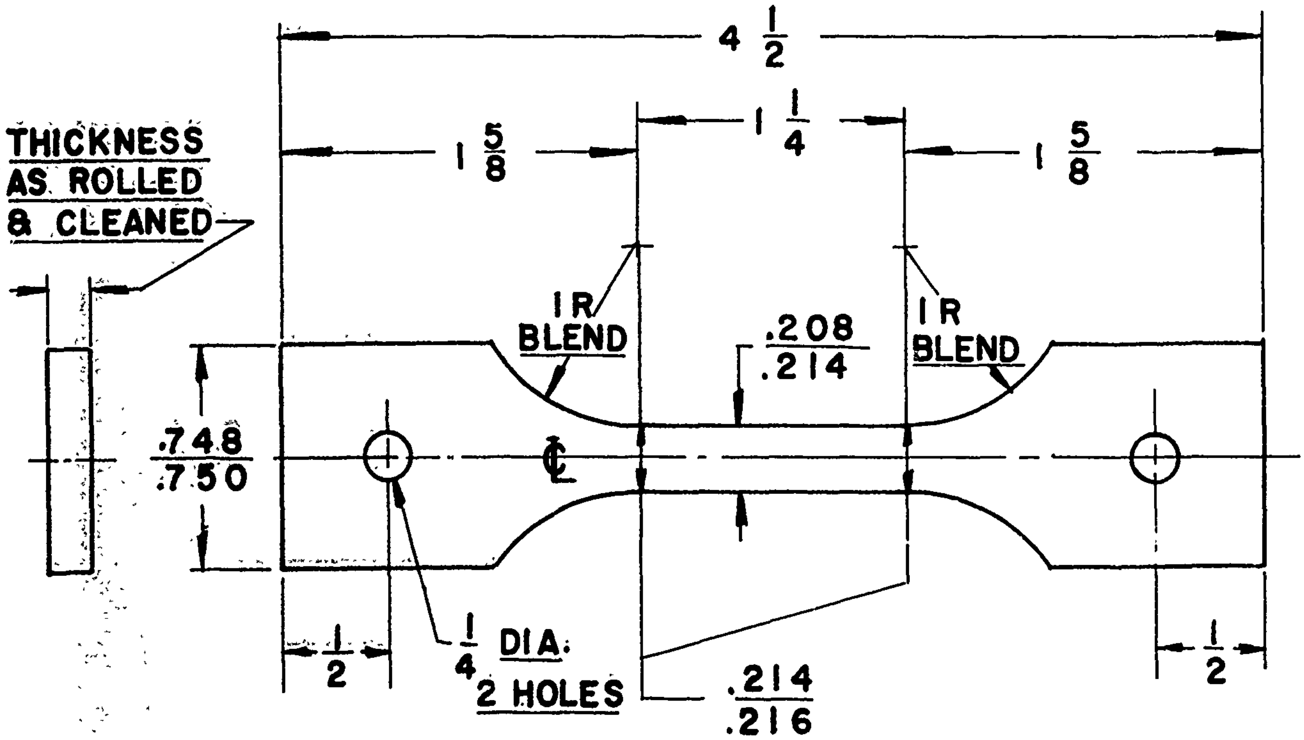
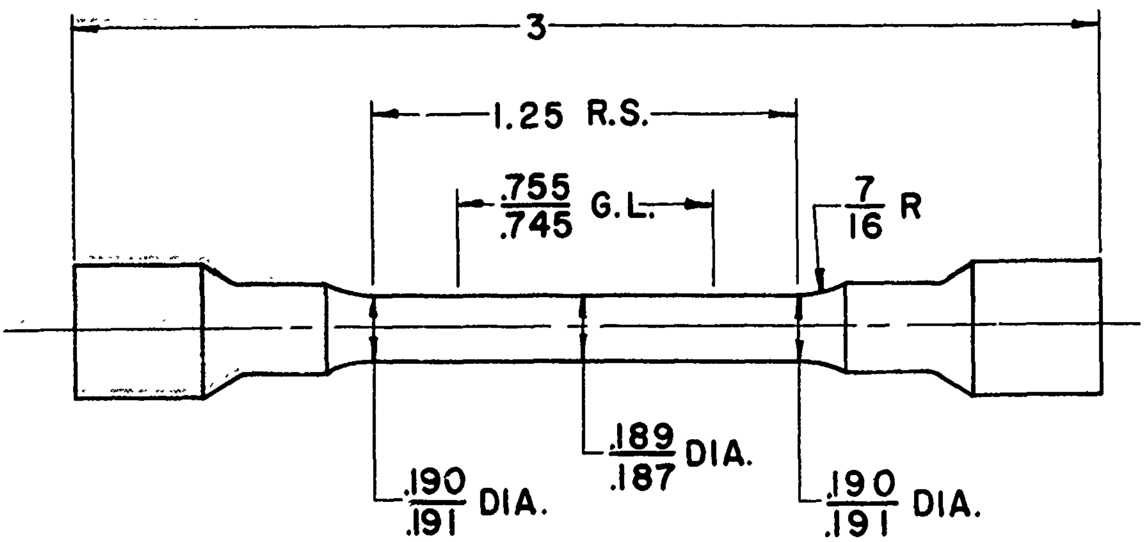


Figure 7 - Grinding Cracks in Machined Sheet Bar



Sheet Tensile Specimen



Round Tensile Specimen

Figure 8 - Tensile Test Specimens

The rectangular sheet tensile specimens were cut from sheets using a circular band saw. Final sizing was accomplished by stack grinding in a contoured fixture. Grinding marks were removed from the edges of the bars by sanding with 4/0 abrasive paper attached to a pneumatic drill head. Premature failures at the locating holes were encountered early in the test program with specimens having a 1/4 inch reduced section and 5/16 inch diameter holes. Reducing both the hole diameter and gage width eliminated this problem.

The majority of sheets were excessively curved after the final roll pass. A flattening procedure was, therefore, established whereby tensile strips were heated, in air, to 1200°F and straightened at light pressures in a hydraulic press between two flat plates.

Data was collected throughout the testing program to determine the effects of surface preparation technique on room temperature tensile properties. Tensile specimens taken from selected hot and hot-warm rolled sheets were tested with surfaces as-rolled, ground and polished, pickled, and electropolished. Pickling of tensile specimens was accomplished by immersion in a solution of concentrated hydrochloric acid at 120-150°F for a time sufficient to remove 1 mil from each surface (usually 30-60 seconds). Electropolished specimens were prepared by immersion in a bath consisting of 10 parts glacial acetic acid and 1 part 60 percent CP Perchloric acid. A time of approximately four minutes was sufficient to remove one mil from each surface when the bath was operated at 110 to 125°F, 21 volts and 3 amps/square inch. This procedure produced a fairly rough surface, ranging from 30 to 90 microinches, due to unequal reaction rates with chromium and MgO.

Tensile tests were performed on a Baldwin Lima Hamilton Universal Testing Machine in accordance with the Materials Advisory Board Standard for Refractory Metals, Procedure 176-M, which specifies a strain rate of 0.5 percent per minute in the elastic range and a rate of 5 percent per minute beyond 0.6 percent offset. For room temperature testing a strain pacer in combination with a Class C snap-on type extensometer was used to control strain rate during test. Strain recordings for all other tests were obtained from a microformer type deflectometer using pre-established cross-head speed settings to obtain the proper strain rates.

For tests conducted below room temperature the tensile specimens and grips were contained within an insulated container filled with acetone and dry ice. A platinum wound resistance furnace was used to heat test specimens for elevated temperature testing. Calibration and attachment of thermocouples were performed according to ASTM E-21 specifications. All elevated temperature tests were made in air using a maximum heating rate and a 2 minute stabilizing dwell at temperature prior to load application.

Oxidation Testing

The equipment used for determining the oxidation behavior of Chrome-30 sheet specimens consisted of an analytical balance suspended above a platinum wound resistance furnace. This equipment, shown in Figure 9, was designed to provide a continuous measurement of weight change throughout the test duration. Sheet test specimens, 1/2 inch square, were contained in a platinum basket in the furnace on a platinum wire attached to one end of the balance beam. All specimens were wet ground to 4/0 abrasive

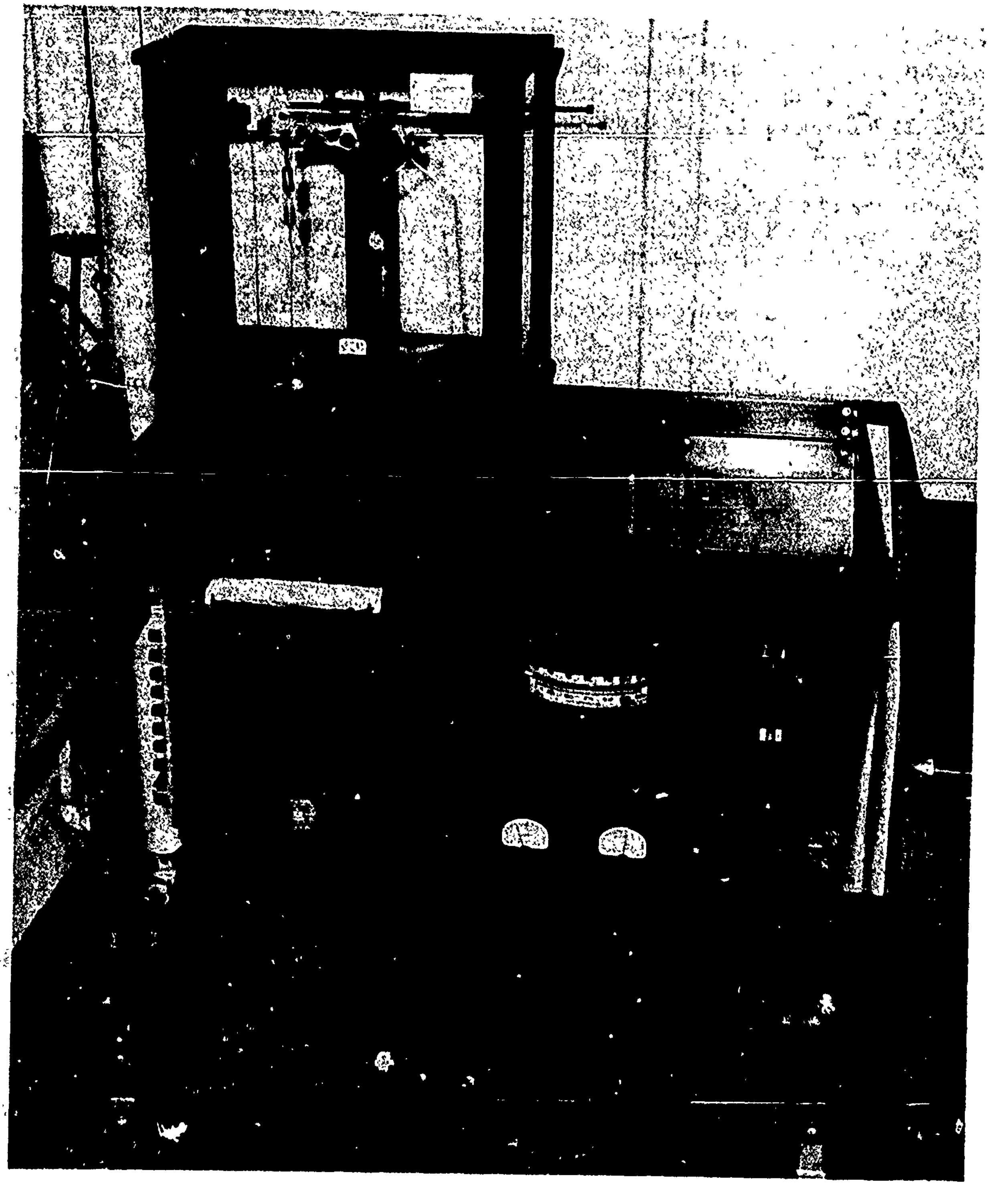


Figure 9 - Thermogravimetric Oxidation Apparatus

paper and dried at 230°F for three hours prior to test. A constant air flow was maintained within the 1-1/2 inch diameter aluminum oxide furnace muffle by introducing dry commercial air at a velocity of 1.0 SCFH. Three samples were exposed for a 24 hour period at each test temperature and the weight gains that were recorded included the weight of any loose scale.

Metallographic Specimen Preparation

Specimens for metallographic examination were mounted in clear lucite and surface ground using a 100/120 grit silicon carbide wheel and water coolant. Following coarse grinding to 4/0 abrasive paper, specimens were coarse polished with an AB Buehler 1585 silk cloth and 1551 polishing alumina No. 2. Final polishing was accomplished with 1 micron chromium oxide on microcloth. Specimens were etched with a solution of 1 part hydrochloric acid to 3 parts glycerin. Repeated etching and final polishing provided satisfactory structures.

EXTRUSION STUDIES

Eighteen billets were extruded at the Aeronautical Systems Division facility to evaluate the effects of variable extrusion procedure and to determine the optimum procedure for producing sheet bar rolling stock.

Extrusion Trials

Extrusions were made at 2000, 2200, and 2400°F using extrusion ratios of 8:1, 10:1 and 12:1 at each temperature. The extrusion trials are summarized in Table 18 in the Appendix and four typical extruded bars are shown in Figure 10. Completely satisfactory extrusions were obtained under all combinations of temperature and ratio. As expected, increased temperature lowered the required pressure for extrusion. The average billet-to-extrusion yield for these 18 billets was 86.5 percent based on the weight of useable extrusion. Typical nose and tail sections trimmed from the extrusion are shown in Figure 11. The co-extruded flame sprayed nickel clad adhered well to the extrusions in all cases. Photographs of typical as-extruded surfaces with the nickel clad intact are shown in Figure 12. Surface finish under the clad was found to range from 150 to 250 microinches RMS. A photograph of a typical surface after clad removal is shown in Figure 13.

Hardness and Density Evaluation

Each of the 18 extrusions was sectioned for evaluation according to the illustration shown in Figure 14. Hardness, microstructure and density specimens were taken from the front, center and rear of each extrusion. The results of these evaluations, which are listed in Table 1, indicate that each extrusion was uniform in hardness and density from front to rear and that there were only minor differences between extrusions performed at different ratios and temperatures. Density and hardness sampling across a center section, transverse to the extrusion direction produced the following results:

Sample Location	1	2	3	4	5	6
Density - g/cm^3	6.43	6.54	6.58	6.58	6.56	6.41
Hardness - R_B	76.5	79.0	79.9	80.0	79.8	76.5

These data reveal a slightly lower hardness and density at the outer edges of the extrusion resulting from the variation of working rate across the flat bar geometry.

Hardness measurements were also made on longitudinal and transverse specimens which were annealed at 1800, 2200, and 2400°F. These data are shown in Figures 15, 16 and 17. The differences in average hardness were small, indicating little if any effect from variation in annealing temperature for the extrusion temperatures and ratios investigated.

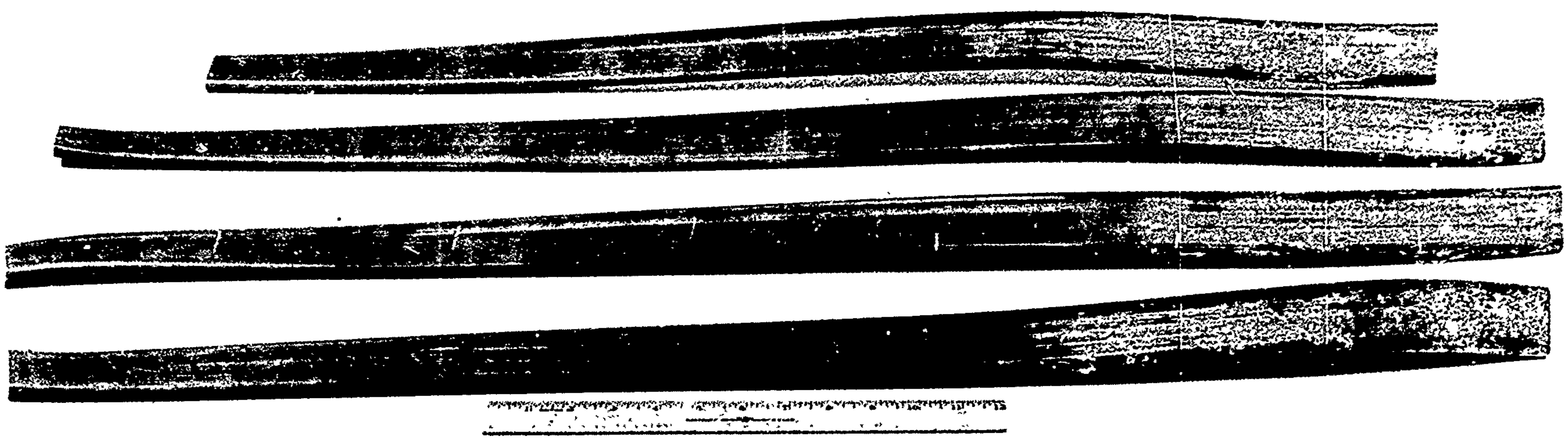


Figure 10 - Typical Extrusions

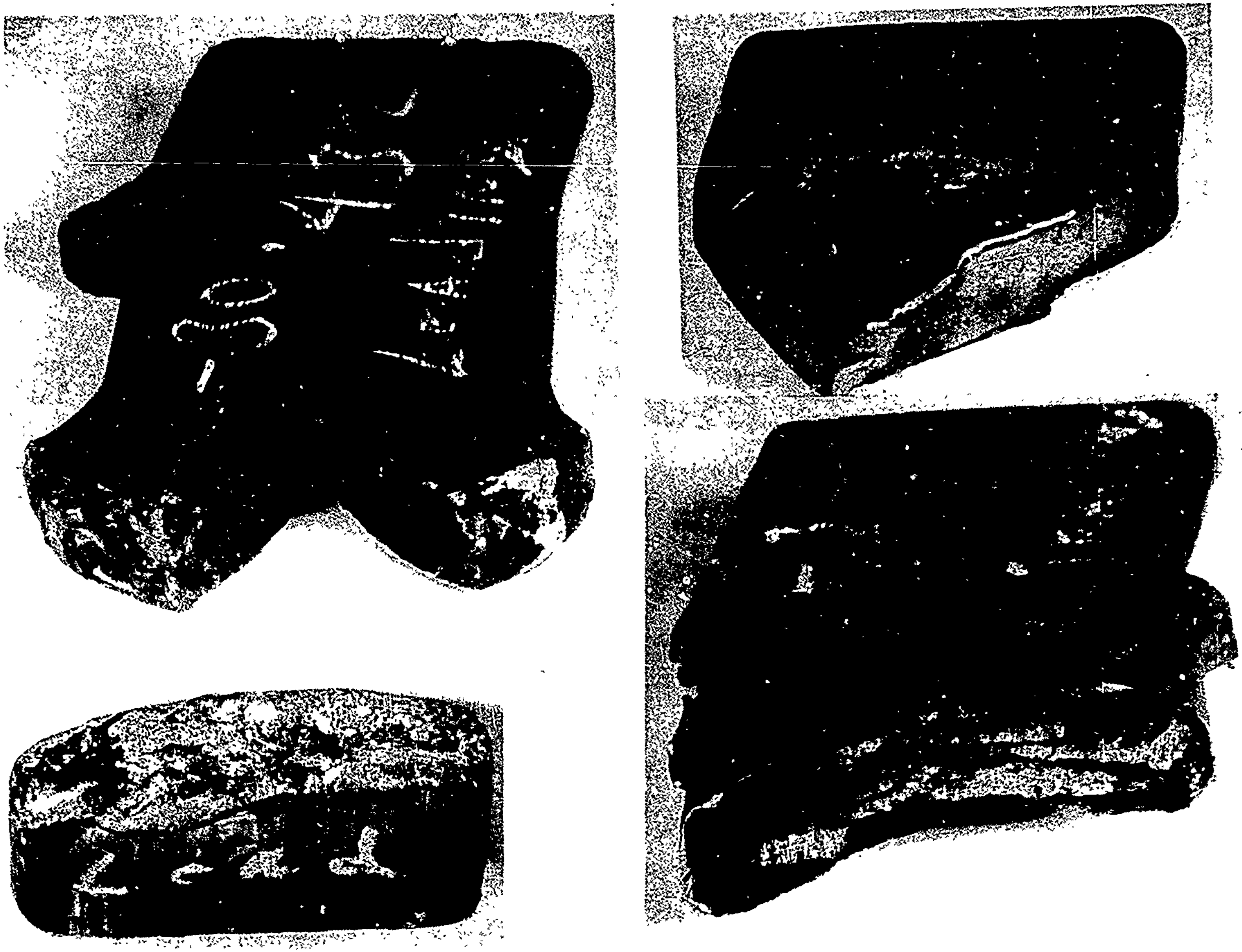
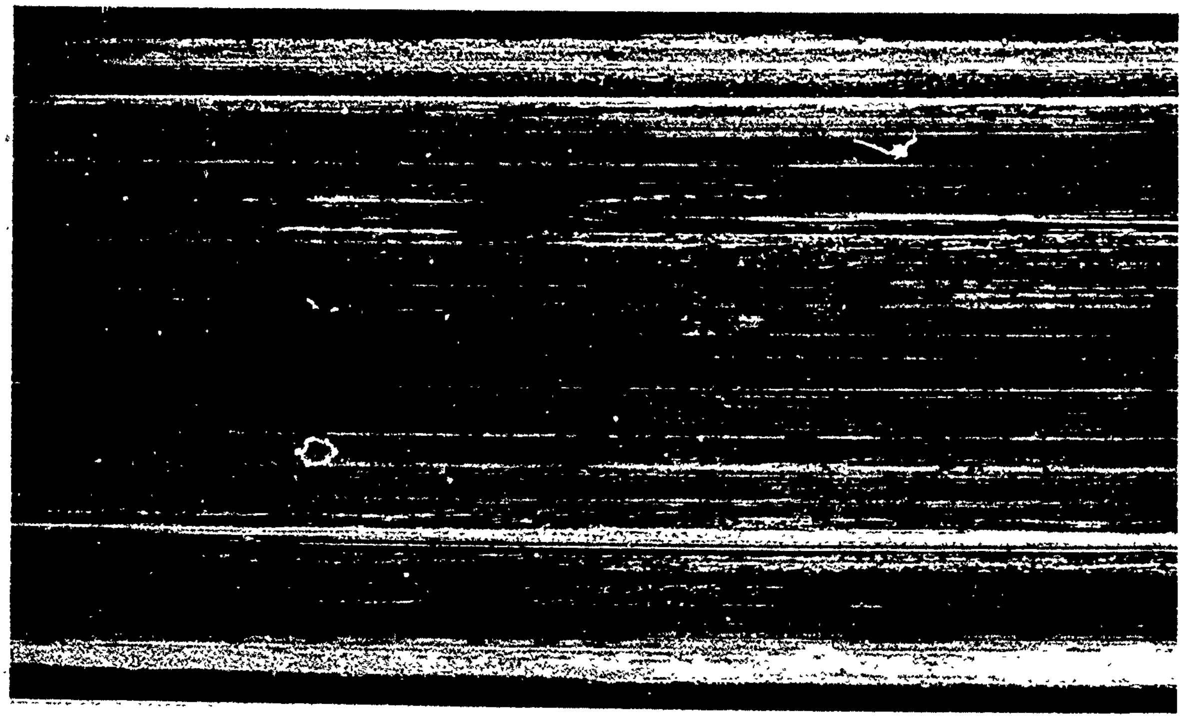
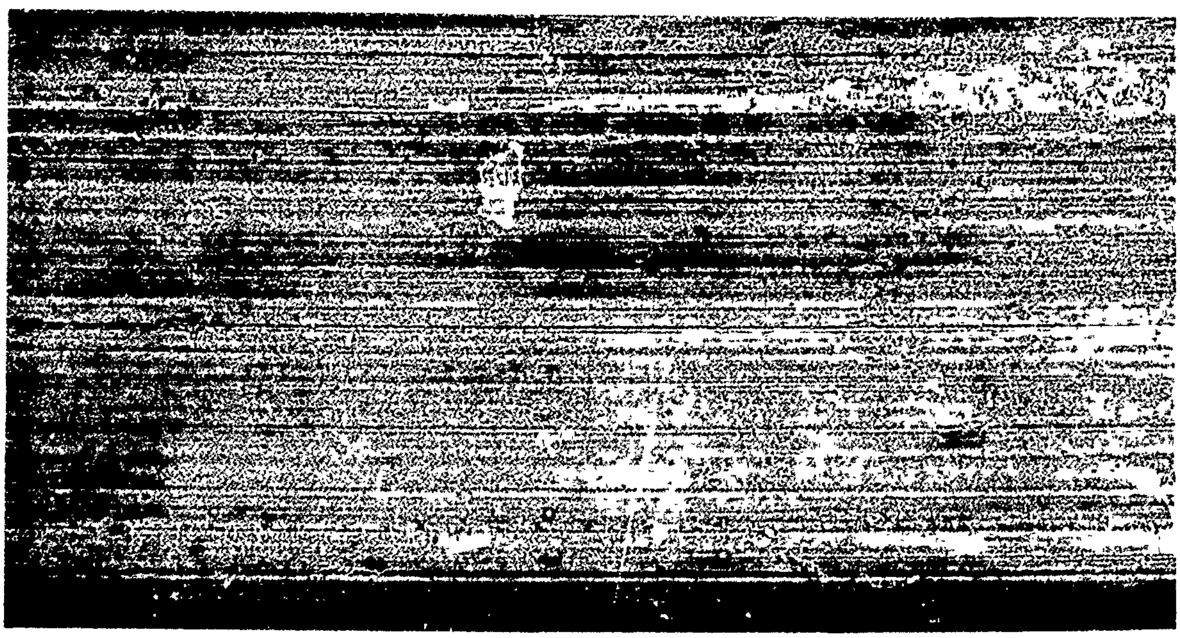


Figure 11 - Typical Nose and Tail Conditions

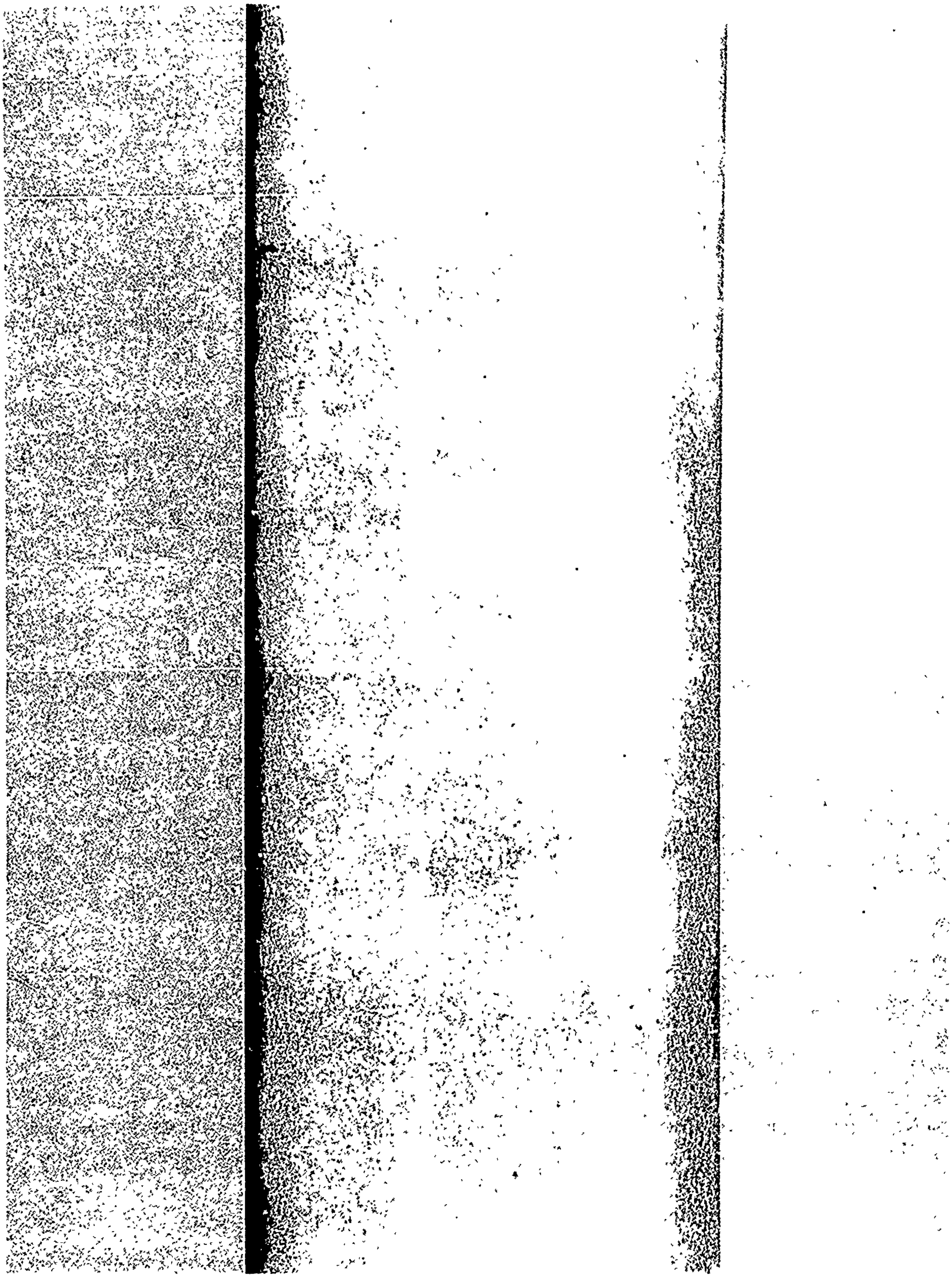


Extrusion 785 2X



Extrusion 784 2X

Figure 12 - A Typical Range of As-Extruded Surface



Extrusion 765 2X

Figure 13 - Surface of Extruded Billet With Clad Removed

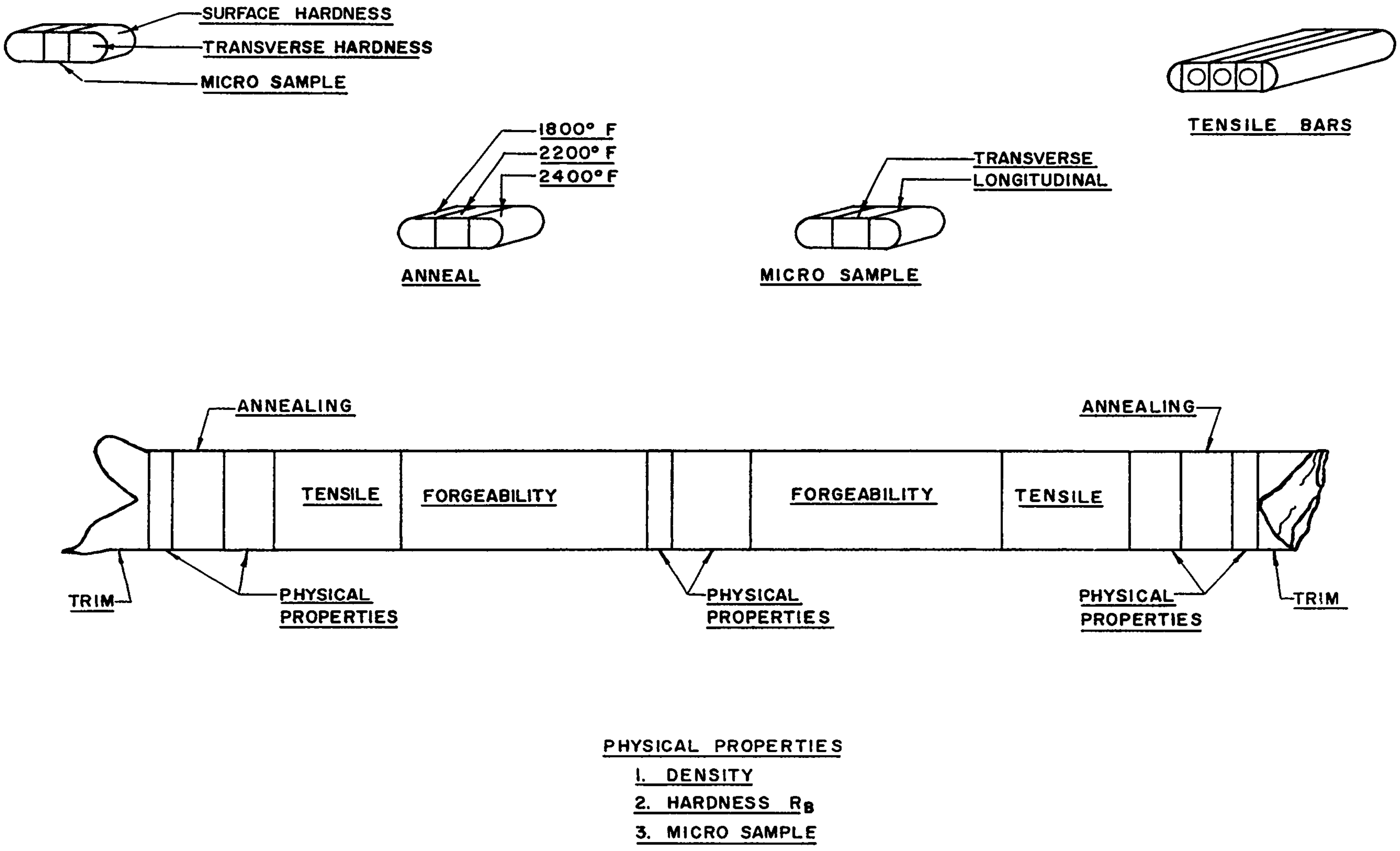


Figure 14 - Disposition of Extrusion Bar

Table 1. Effect of Extrusion Temperature and Ratio on the Hardness, Density and Percent Yield of Chrome-30 Extrusions

Extrusion Number	Extrusion Temperature, F	Extrusion Ratio	Extrusion Yield, Percent	Extrusion Density g/cc			Hardness Rockwell B		
				Front	Center	Back	Front	Center	Back
764	2200	10:1	80.3	6.55	6.56	6.54	75.5	74.8	77.7
765	2000	9.6:1	85.1	6.60	6.54	6.56	78.1	77.8	75.3
766	2200	"	87.7	6.55	6.56	6.57	76.4	75.7	76.0
767	2000	"	87.3	6.60	6.55	6.58	78.7	77.3	77.2
768	2400	"	86.2	6.57	6.55	6.57	77.6	75.2	76.1
769	2400	10:1	86.7	6.55	6.56	6.57	74.5	75.2	74.1
780	2000	12:1	87.0	6.54	6.54	6.55	78.0	77.0	80.0
781	2000	12:1	80.4	6.56	6.55	6.55	78.0	78.0	74.0
782	2000	8:1	88.0	6.52	6.54	6.60	78.0	77.0	78.0
783	2000	"	88.4	6.54	6.54	6.52	80.0	79.0	78.0
784	2200	"	89.4	6.53	6.55	6.52	78.0	78.0	75.0
785	2200	"	89.5	6.54	6.55	6.55	77.0	77.0	79.0
786	2400	"	81.1	6.56	6.59	6.59	79.0	79.5	81.0
787	2400	"	87.2	6.55	6.56	6.55	80.0	80.0	79.0
808	2400	12:1	89.0	6.53	6.57	6.55	75.9	77.0	76.3
809	2400	"	88.0	6.56	6.55	6.55	80.2	79.0	77.2
810	2300	"	88.8	6.55	6.55	6.54	75.3	78.2	74.5
811	2200	"	88.2	6.56	6.57	6.54	79.3	78.6	78.7

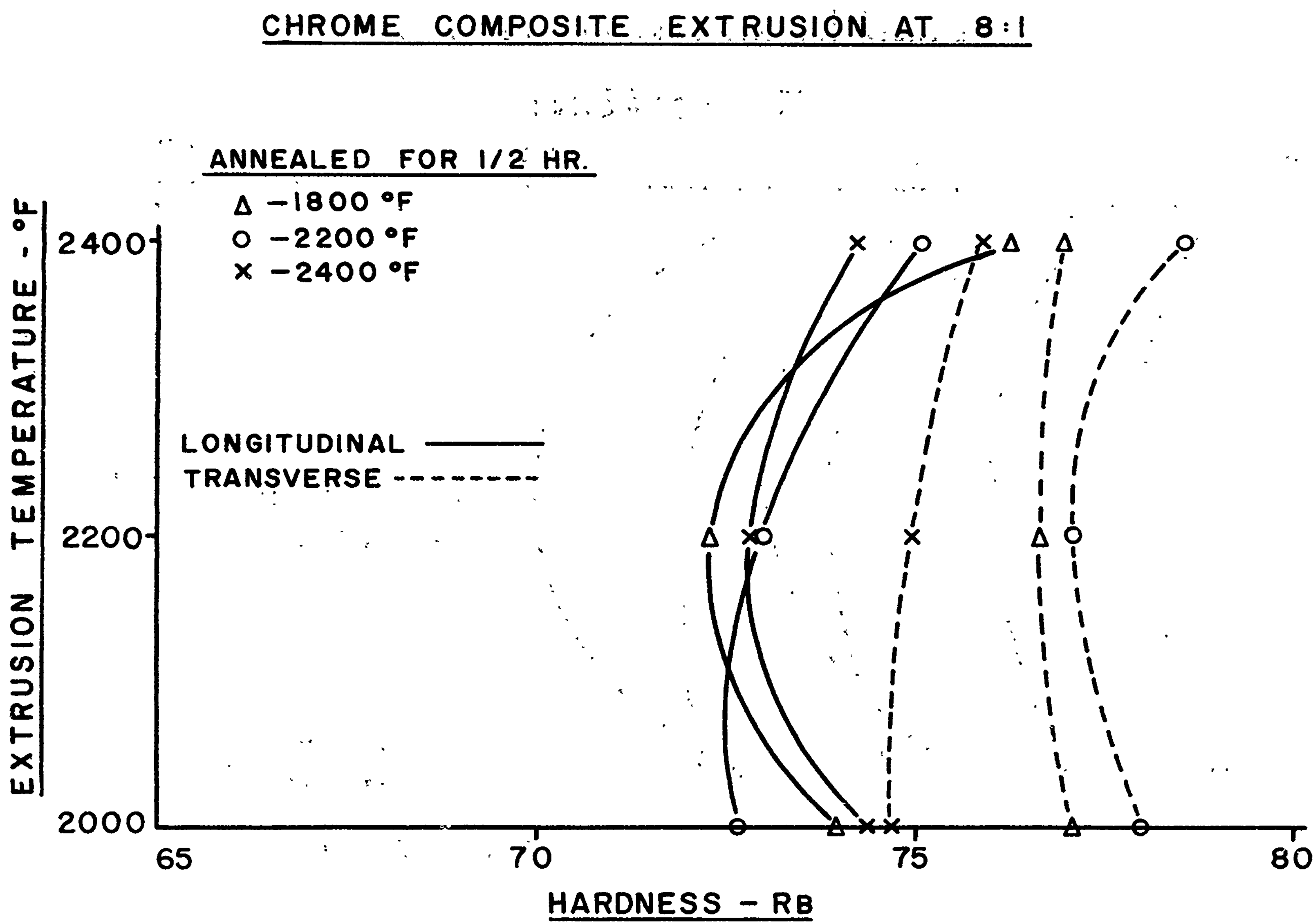


Figure 15 - Annealing Response 8:1 Extrusions

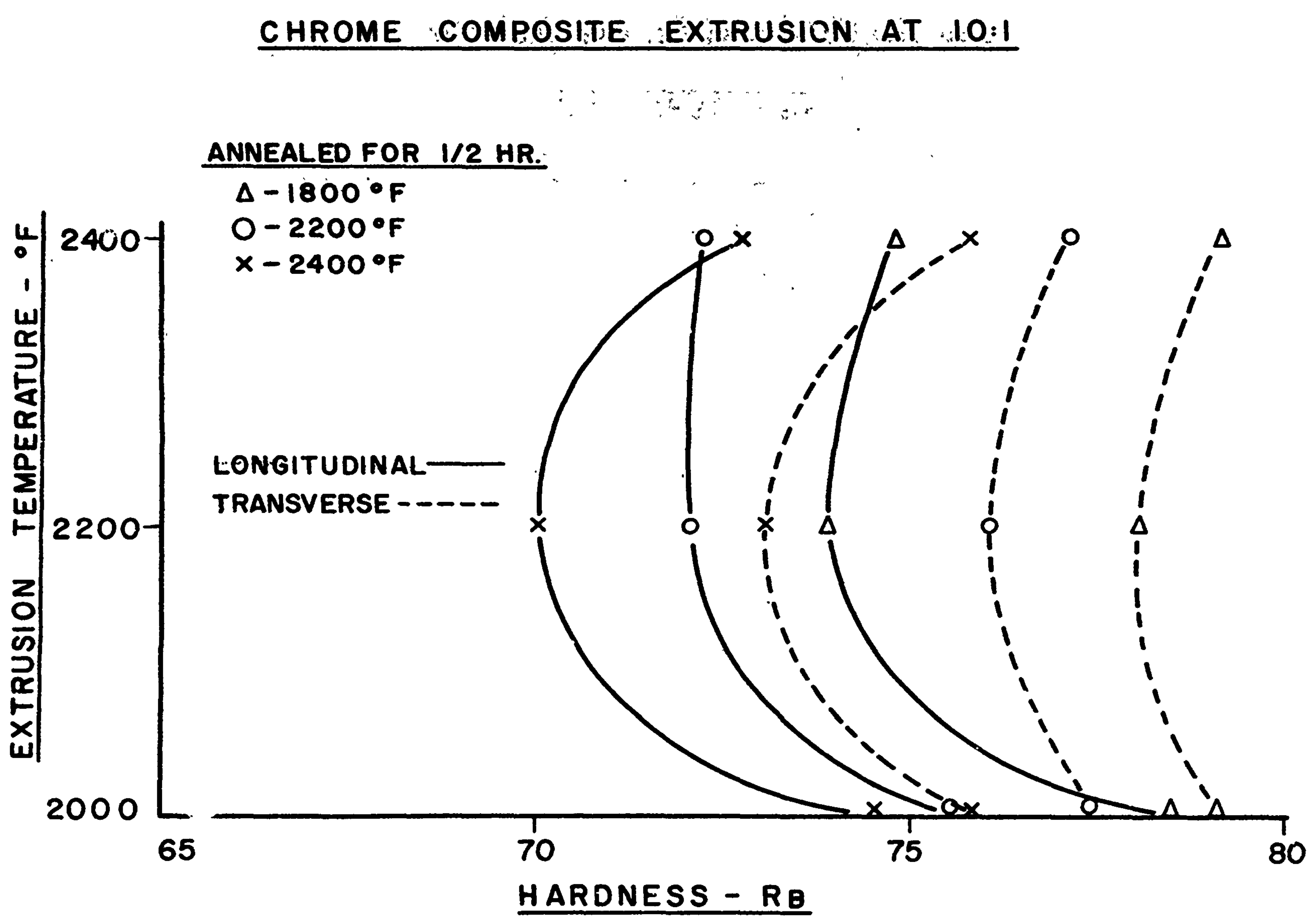


Figure 16 - Annealing Response 10:1 Extrusions

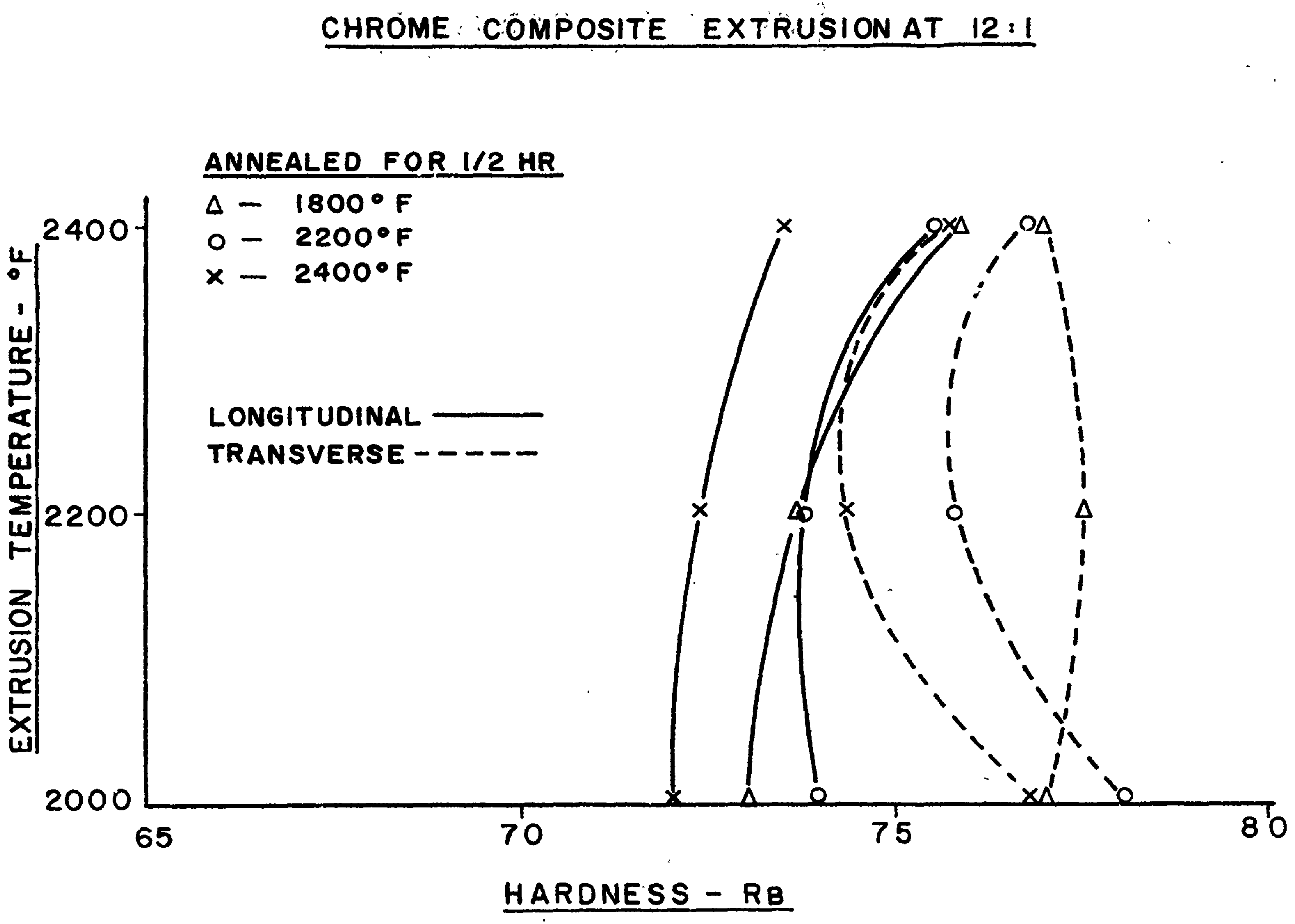


Figure 17 - Annealing Response 12:1 Extrusions

Microstructure Evaluation

Examination of four specimens from each extrusion revealed that microstructures were independent of extrusion temperature and ratio. All structures appeared fully recrystallized in the as-extruded condition and no differences were detected in microstructures taken from various locations along the length of each extrusion. Structures of specimens annealed at 1800, 2200 and 2400°F for one hour were identical to the as-extruded structures with the exception of a slight grain growth observed for the higher annealing temperature. The photomicrographs shown in Figure 18 represent typical microstructures of extrusions made at 2000°F.

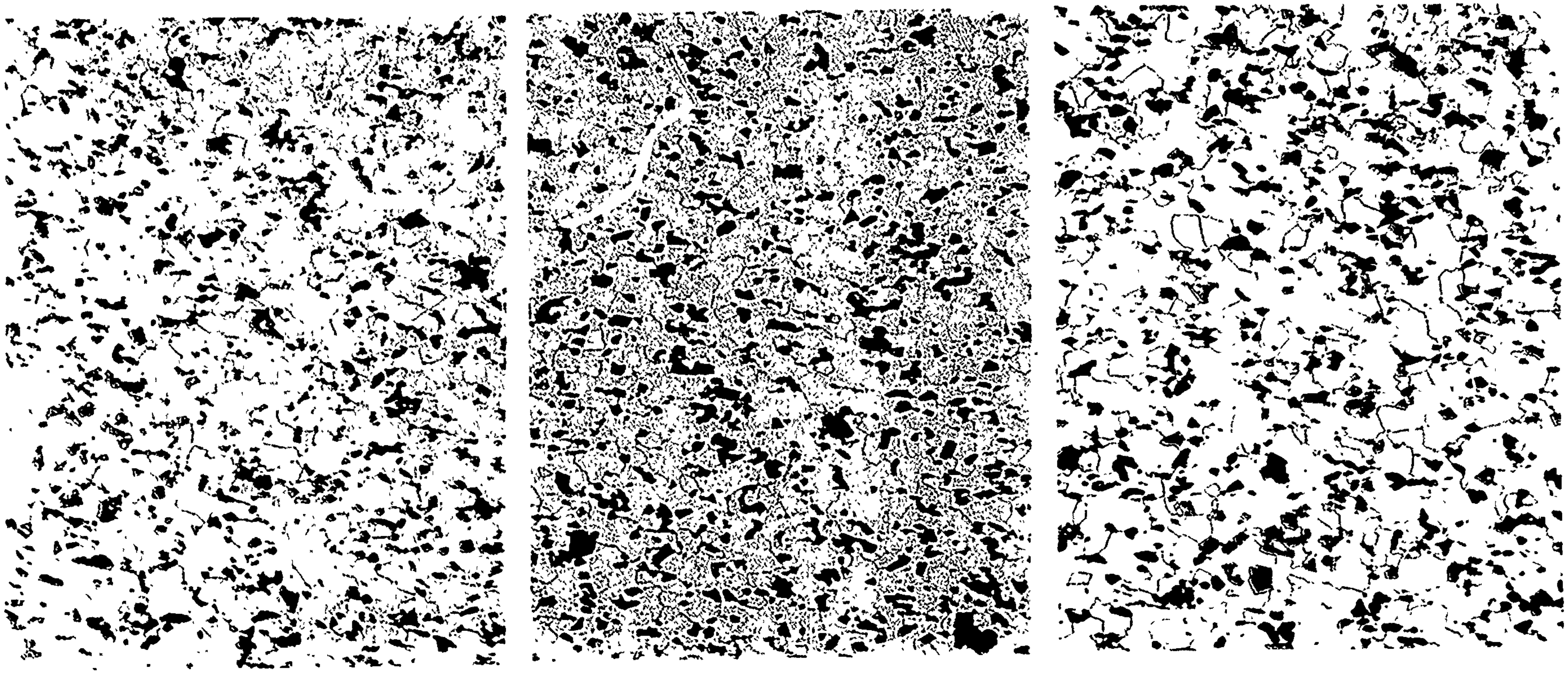
In the course of evaluation several non-typical structures were observed. Photomicrographs (a) and (b) in Figure 19 show, at two magnifications, an oxide stringer which was found close to the surface of one extrusion. Another unusual stringering effect is shown in photomicrograph (c). The porous area shown in photomicrograph (d) was found in a section taken from the trailing end of an extrusion.

Room Temperature Tensile Properties

Six tensile bars were taken from each extrusion to provide an evaluation of extrusion temperatures and ratios. Three tensile bars from each were tested in the as-extruded condition and three were vacuum annealed in 1800°F for two hours prior to test. The results of these tests are given in Table 2.

The entire population of values for strength and elongation are in close accord for all extrusions. The average elongation was 23 percent and the average ultimate strength was 48,000 lb./sq. in. Standard deviation (sigma) on the total population of values for elongation was 1.67 percent. This means that of the entire set of values, regardless of extrusion temperature or ratio, 99 percent would fail within plus or minus 5 percent of 23 percent elongation. Two of the most consistant sets of billets, the 10:1 extrusion at 2000 and 2200°F, had an average elongation of 23.2 percent and 24.1 percent. While the ultimate strength in the as-extruded condition varied slightly, the yield strengths had a somewhat wider scatter. The principal effect of annealing was to slightly lower the yield strength. Percent elongation was not affected. The typical triangular fracture experienced on all bars is shown in Figure 20 which compares tensile bars before and after test. The comparatively low values of reduction in area reported in Table 2 are typical of composite materials which exhibit uniform elongation within the gage length in contrast to conventional necking.

The ductility of completely strain free chrome composite is of particular interest when compared to prior work, which has demonstrated that cold worked pure chromium possesses ductility at room temperature but suffers loss of all ductility upon recrystallization. The evaluation of some excess stock from extrusion #780 was undertaken to determine the ductility of completely strain free composite. Three sample bars were heated at 2900°F for 1/2 hour and then furnace cooled at 1000 degrees per hour. The tensile data for the material, before and after heat treatment, are as follows:



8:1 Ratio

@2000 F

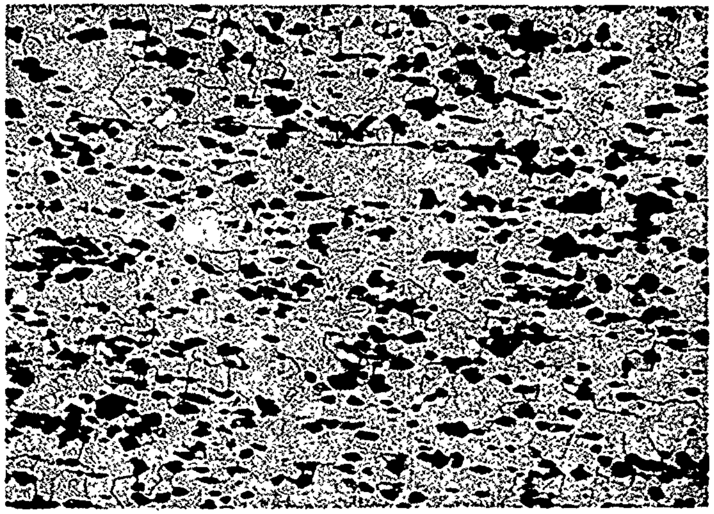
10:1 Ratio

@2000 F

12:1 Ratio

@2000 F

Figure 18 - Typical Transverse Microstructures of As-Extruded Chrome-30 (200X)



0-625 200X

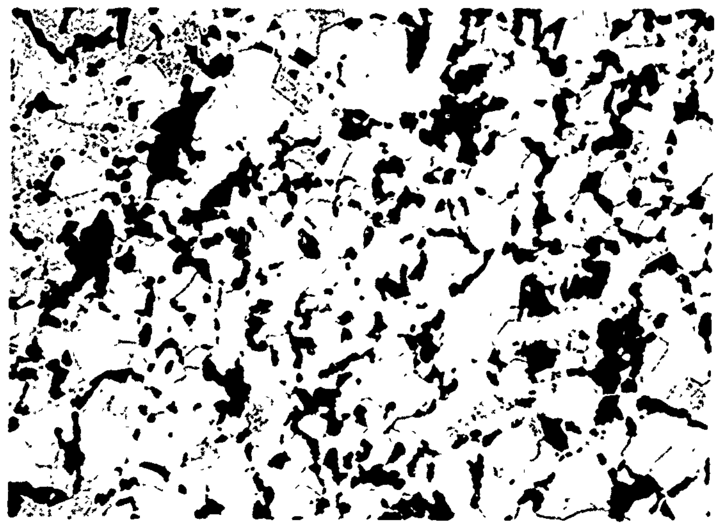
a

0-625 1000X

b

0-669 200X

c



Extrusion 808 200X

d

Figure 19 - Non-Typical Effects in 12:1 Extruded Bar

Table 2. Effect of Extrusion Temperature and Ratio on the Room Temperature Tensile Properties of As Extruded and Annealed Chrome-30[(a)]

Extrusion Ratio	Extrusion Temperature, F	0.2% Offset Yield Strength, 1000 PSI	Ultimate Tensile Strength, 1000 PSI	Elongation in 3/4 Inch, Percent	Reduction in Area, Percent
		As Extruded			
8:1	2000	28.6	48.8	22.1	14.0
	2200	26.6	47.7	21.5	14.1
	2400	25.7	47.6	21.3	12.7
10:1	2000	31.1	49.3	24.1	13.8
	2200	30.8	47.4	24.5	14.0
	2400	24.5	45.9	23.5	14.6
12:1	2000	29.8	47.9	20.8	13.9
	2200	26.1	47.0	22.2	13.8
	2400	24.4	48.0	22.9	15.2
		Vacuum Annealed Two Hours At 1800°F			
8:1	2000	28.1	47.7	24.2	13.0
	2200	24.4	46.4	24.3	14.0
	2400	26.0	47.4	22.0	13.4
10:1	2000	27.0	47.6	22.3	14.3
	2200	25.6	46.4	23.7	14.0
	2400	24.5	45.8	23.6	14.6
12:1	2000	28.2	47.1	22.5	14.1
	2200	24.2	45.5	22.0	13.9
	2400	24.4	47.4	23.7	15.4

(a) Values listed are the average of 3 tensile tests per MAB-176-M

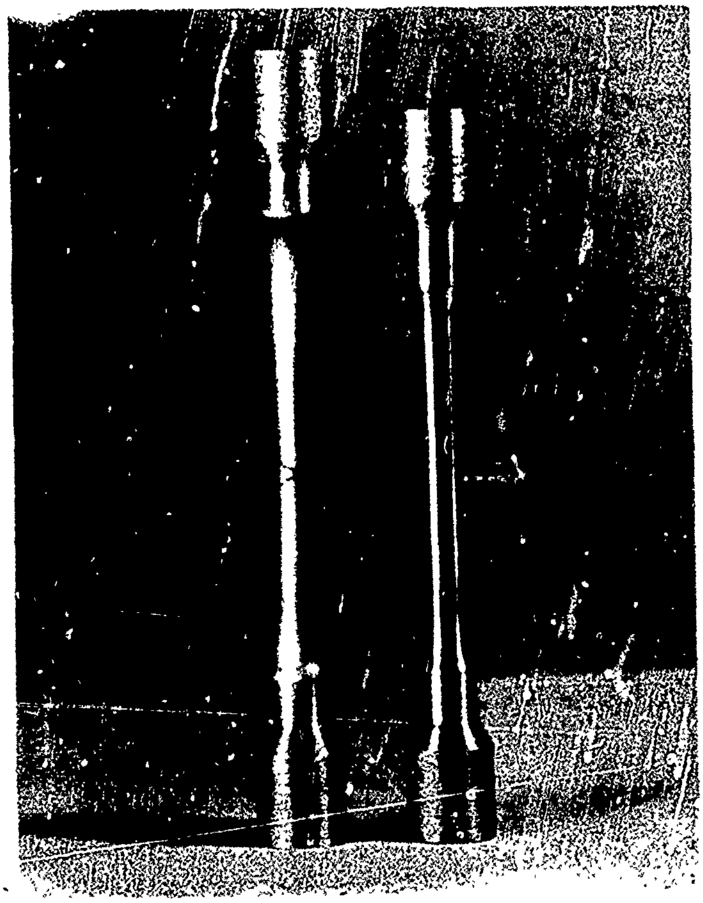


Figure 20 - Typical Tensile Bar Before & After Test

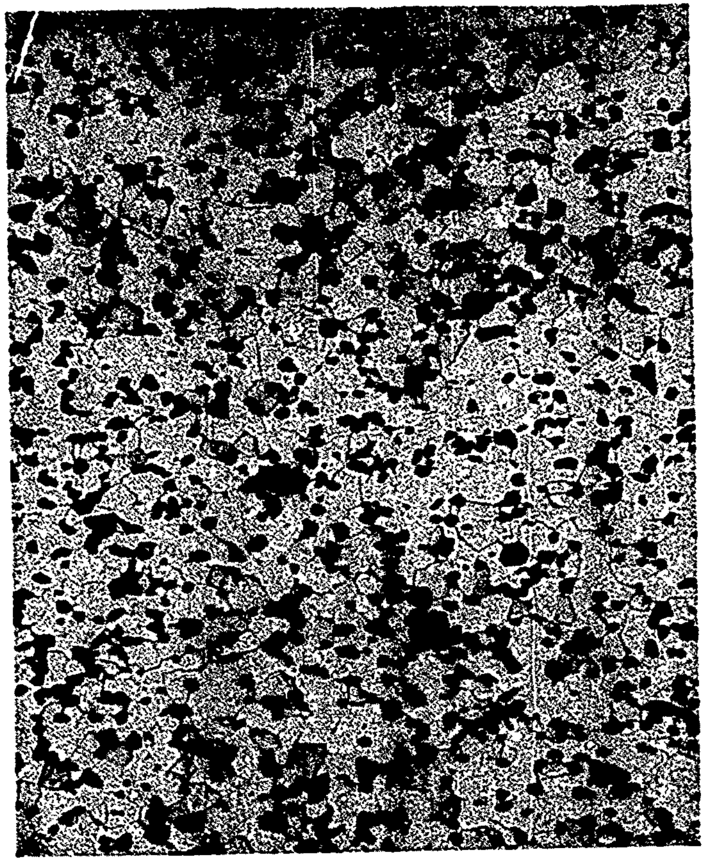
	Yield Strength, 1000 PSI	Ultimate Strength, 1000 PSI	Elongation in 3/4 Inches, Percent	Reduction in Area Percent
As Extruded	27.5-30.0	48.0-49.0	25.0-26.7	13.6-16 0
After 1/2 hour @2900°F	21.0-22.0	40.0-42 0	13.5-13.6	7.1-7.2

The microstructures of the annealed and as-extruded material are shown in Figure 21. The high temperature anneal caused the MgO particles to spherodize and the matrix grain size to increase. The lower strengths shown by the annealed bars were undoubtly due to these structural changes. Limited impact testing indicated that the impact transition temperature is not affected by the anneal at 2900°F.

Ductile-to-Brittle Transition Behavior

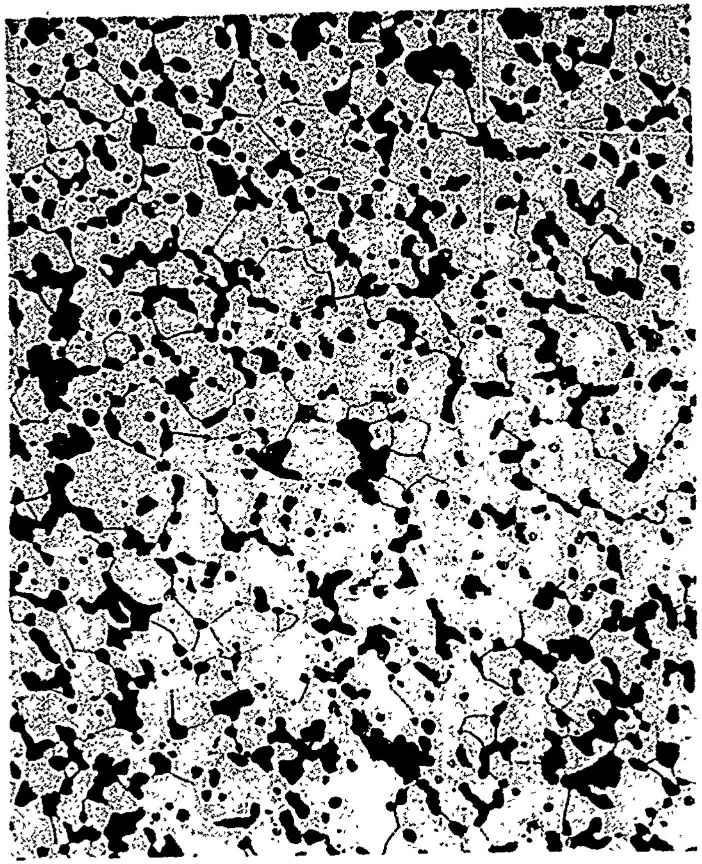
Sufficient material remained from the 12:1 extrusion to allow for some further work under Bendix sponsorship to study the ductile-to-brittle tensile and impact transition of extruded Chrome-30. The effect of low temperatures on tensile properties is shown in Figure 22, which indicates a transition temperature of approximately 10°F. The yield and ultimate strength are shown to have increased in a normal manner with decreasing temperatures.

Unnotched Izod bars of as-extruded material were fractured to determine the impact transition temperature. Figure 23 illustrates that the impact strength exceeded the 30 foot-pound hammer capacity at 475°F and above. Typical ductile and brittle impact behavior is illustrated in the photograph of Figure 24. The rate of impact was 11 ft./sec. for all tests.



0-668 200X

a



0-840 200X

b

Figure 21 - Microstructure of Extruded & Strain Free Chrome-30

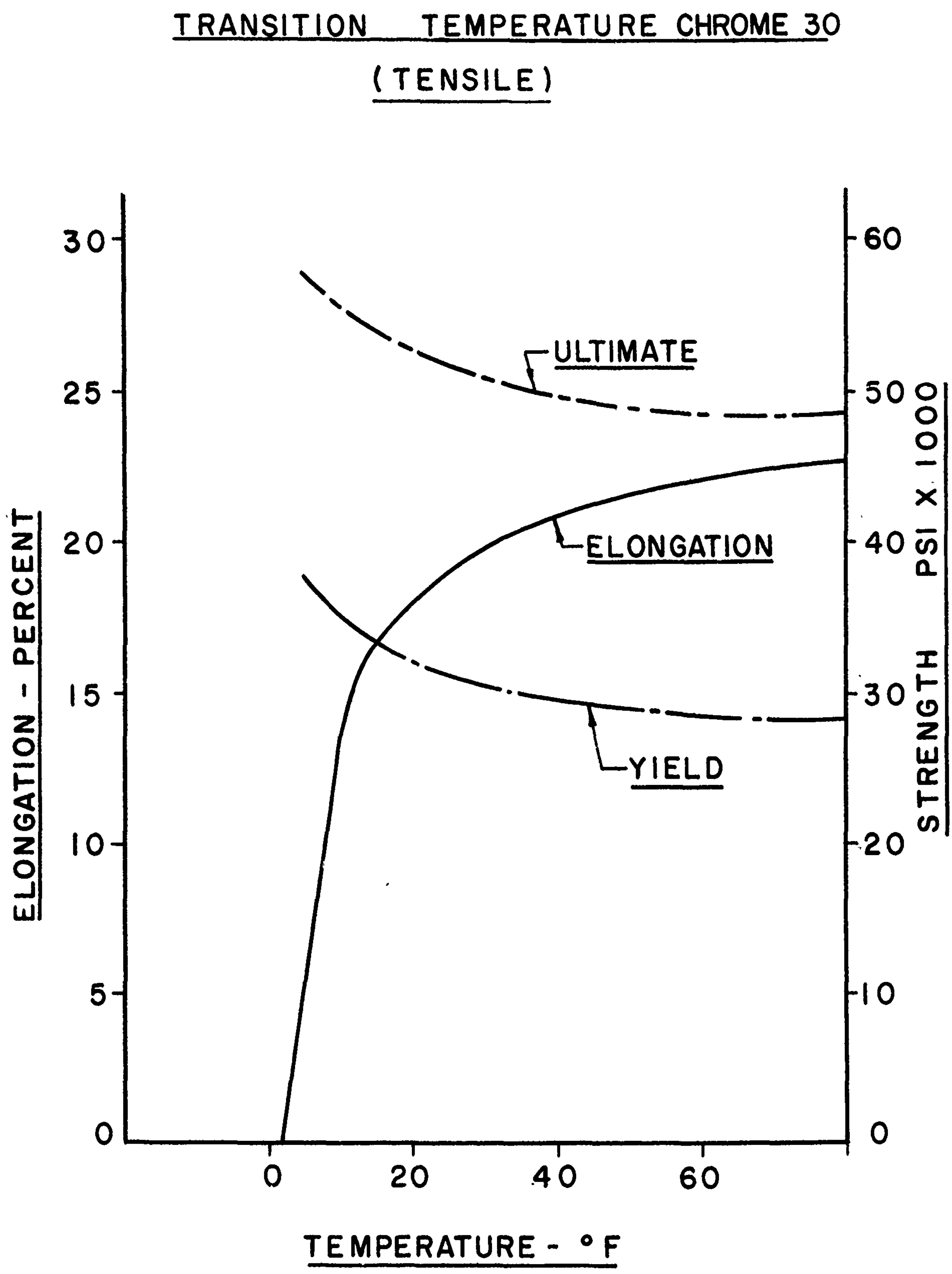


Figure 22 - Ductile-Brittle Tensile Transition of Extruded Chrome-30

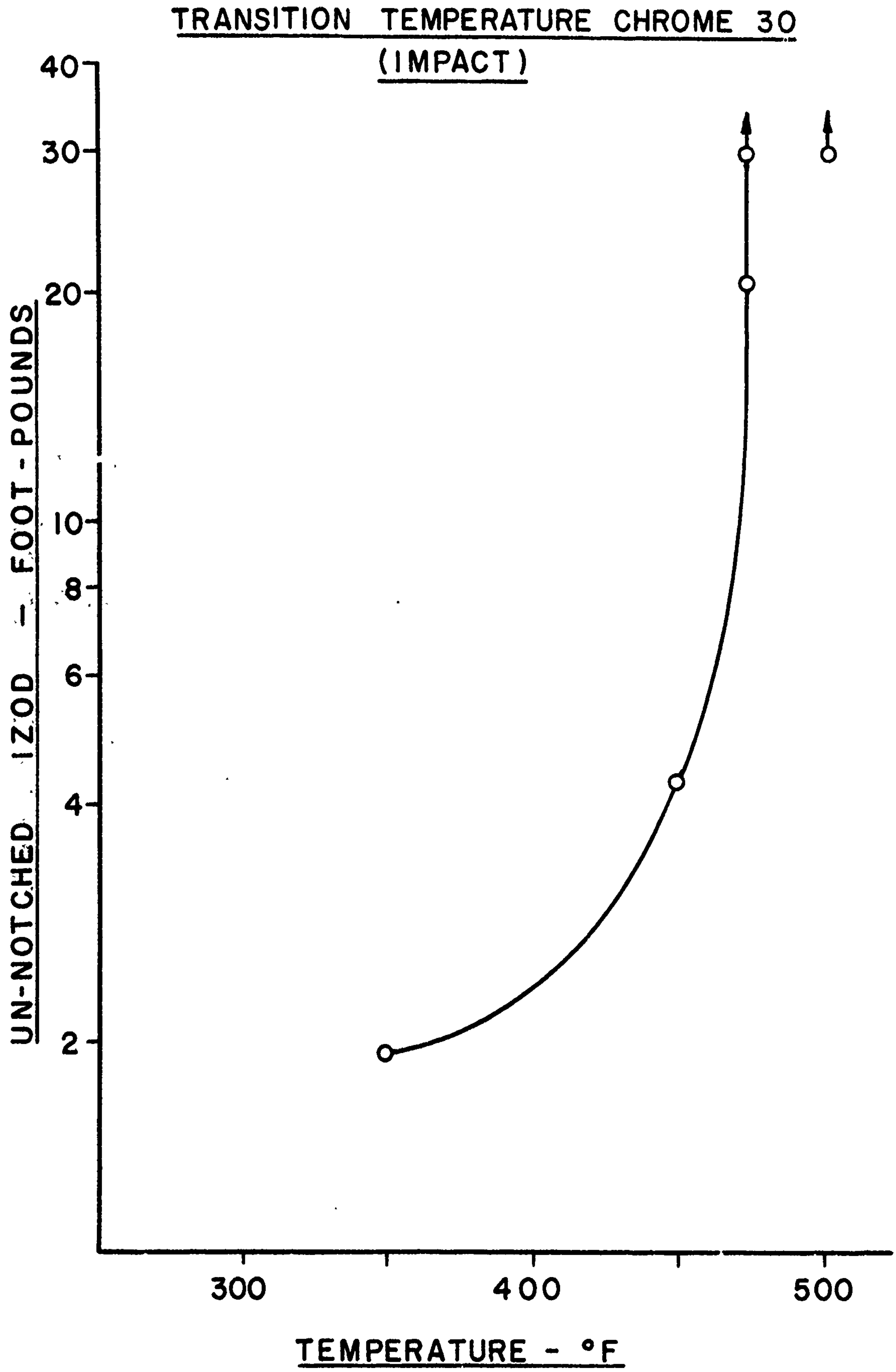


Figure 23 - Ductile-Brittle Impact Transition of Extruded Chrome-30

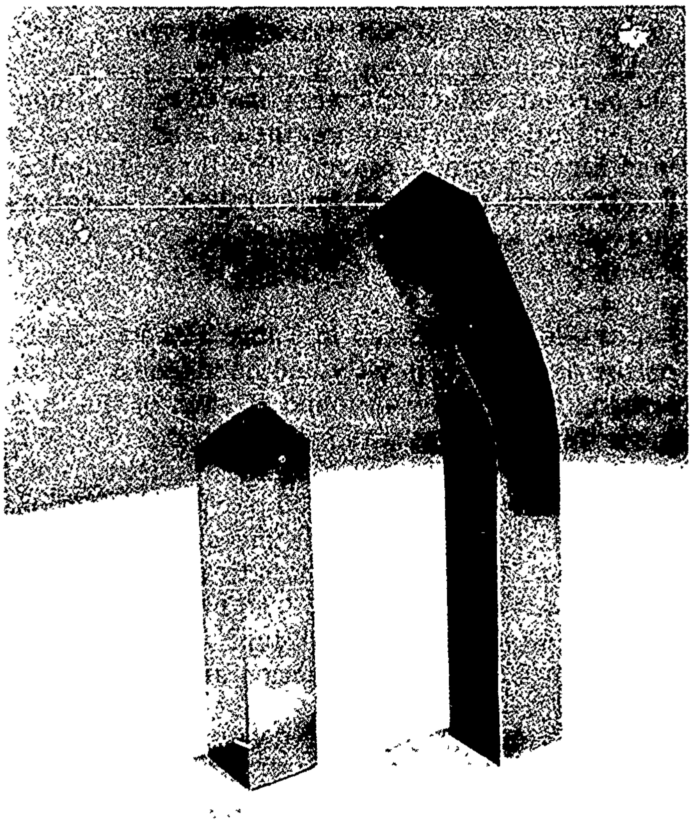


Figure 24 - Impact Bars - Brittle and Ductile Behavior

FORGING STUDY

Press forging was used to provide preliminary data on the hot and warm workability of extruded Chrome-30 and to further evaluate extrusion procedures. Twelve flat forging samples approximately 1 inch by 1-1/2 inches were cut from each of the 18 extrusions. As-extruded and annealed samples were upset-forged to 20, 40, and 60 percent reduction at 400, 2000, 2200 and 2400°F.

Hot Forging Results

It was found that the low density extrusion edges, discussed previously, significantly retarded forgeability. Samples which initially contained a portion of the half round extrusion edge developed edge cracks at low reductions. Satisfactory forgings were obtained when these edges were completely removed.

Equivalent forging response was obtained from all extrusions throughout the spectrum of extrusion ratios and temperatures. Similar forging characteristics were also obtained for both as-extruded and annealed samples. At the three hot forging temperatures selected, the 20 and 40 percent reductions were successful without exception. Reductions of 60 percent, however, resulted in random external and internal cracks independent of extrusion procedure and forging temperature. The three samples shown in Figure 25, from an 8:1 - 2200°F extrusion, represent typical 2200°F forgings at the three reductions. Typical edge splits which resulted from 60 percent reductions at 2400°F are shown in Figure 26.

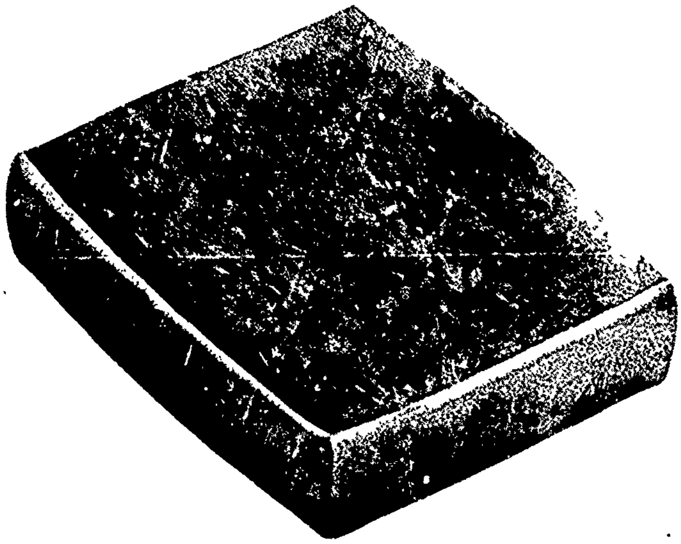
The selected forging trials summarized in Table 3 show the effect of forging temperature and reduction on the as-forged density and hardness. An increase in hardness and density with increasing reduction was found at each of the three forging temperatures. This apparent work hardening was evident in the microstructures of samples forged 40 percent and 60 percent. The typical grain distortion shown in photomicrographs (a) and (b) of Figure 27 resulted from 60 percent reductions at 2000 and 2400°F respectively. Internal cracks were detected in the microstructure of several samples which were forged 60 percent. Typical crack patterns in a 2400°F forging are shown in photomicrographs (c) and (d) of Figure 27. The best forging response was obtained from the 10:1 - 2000°F extrusion. Only one of four samples from this extrusion contained internal cracks after a 60 percent reduction at 2400°F.

Warm Forging Results

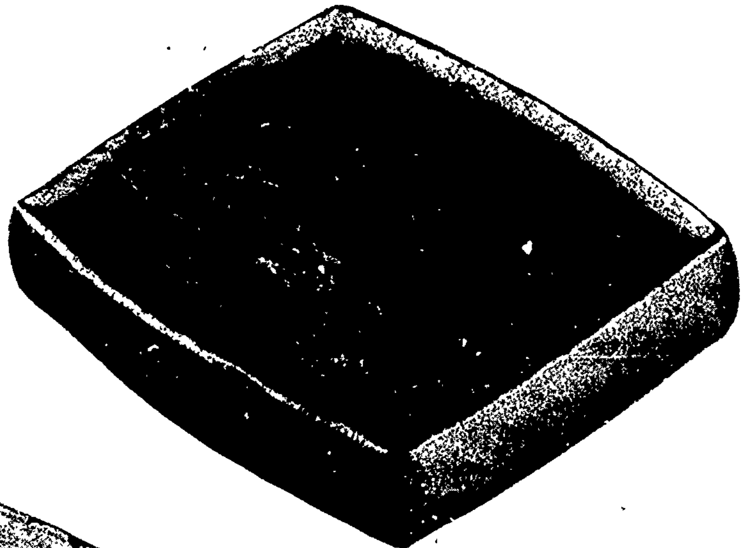
A forging temperature of 400°F produced good forging response. Typical forged samples are shown in Figure 28. Sample b and c, taken from a 10:1 - 2400°F extrusion, were reduced 20 and 40 precent respectively at 400°F without edge splits. The low density extrusion edges were not removed from sample a and, as a result, an edge split developed when the sample was forged 20 percent at 400°F.

Recrystallization Behavior

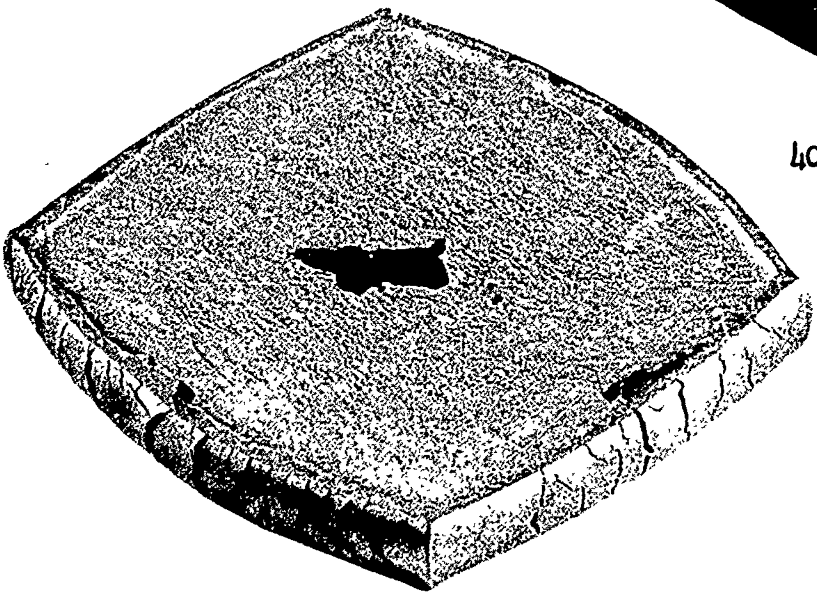
A recrystallization temperature of 1700°F was established for extruded Chrome-30 warm forged 50 percent at 500°F. Microstructural changes resulting from 1/2 hour



a
20% 1X



b
40% 1X



c
60% 1X

Figure 25 - Various Forge Reductions on 8:1 Extrusion

Table 3. Effect of Forging Temperature and Reduction on the Density and Hardness of Selected Extrusion Samples

Extrusion Number	Extrusion Ratio	Extrusion Temperature, F	Sample Condition(a)	Intended Forging Reduction, Percent	Actual Reduction, Percent	Forging Temperature, F	Forged Density, g/cm^3	Forged Hardness, Rockwell B(b)
785	8:1	2200	A	20	19.7	1800	6.53	96.8
785	8:1	2200	A	40	42.0	1800	6.55	100.5
784	8:1	2200	A	60	57.6	1800	6.51	99.7
785	8:1	2200	A	20	23.9	2100	6.50	89.0
785	8:1	2200	A	40	32.5	2100	6.51	100.0
784	8:1	2200	A	60	55.1	2100	6.58	101.5
785	8:1	2200	A	20	22.1	2400	6.50	85.5
785	8:1	2200	A	40	35.0	2400	6.54	98.3
784	8:1	2200	A	60	53.7	2400	6.59	102.2
764	10:1	2200	A	60	40.6	2400	6.60	102.5
765	9.6:1	2000	A	60	40.7	2400	6.58	103.6
766	9.6:1	2200	A	60	40.6	2400	6.58	101.9
767	9.6:1	2000	A	60	44.0	2400	6.60	101.9
768	9.6:1	2400	A	60	44.8	2400	6.57	102.4
769	10:1	2400	A	60	48.0	2400	6.61	104.4
764	10:1	2200	AE	60	53.0	2400	6.57	102.3
765	9.6:1	2000	AE	60	53.2	2400	6.64	101.8
766	9.6:1	2200	AE	60	54.4	2400	6.59	102.0
767	9.6:1	2000	AE	60	57.4	2400	6.66	100.9
768	9.6:1	2400	AE	60	53.9	2400	6.69	101.6
769	10:1	2400	AE	60	56.5	2400	6.66	102.4
780	12:1	2000	AE	60	51.0	2400	6.59	102.2
781	12:1	2000	A	60	48.1	2400	6.59	101.5
783	8:1	2000	AE	60	52.6	2400	6.60	102.7
782	8:1	2000	A	60	53.6	2400	6.59	102.3
784	8:1	2200	AE	60	52.3	2400	6.58	101.5
786	8:1	2400	AE	60	54.0	2400	6.60	102.3
786	8:1	2400	A	60	51.7	2400	6.60	103.3
808	12:1	2400	A	60	54.6	2400	6.60	95.7
809	12:1	2400	AE	60	48.5	2400	6.57	101.3
810	12:1	2300	A	60	51.2	2400	6.59	93.0
811	12:1	2200	AE	60	49.4	2400	6.56	102.2

(a) A - Vacuum annealed for 2 hours at 1800 F. AE - As Extruded.

(b) Major load applied for 5 seconds.

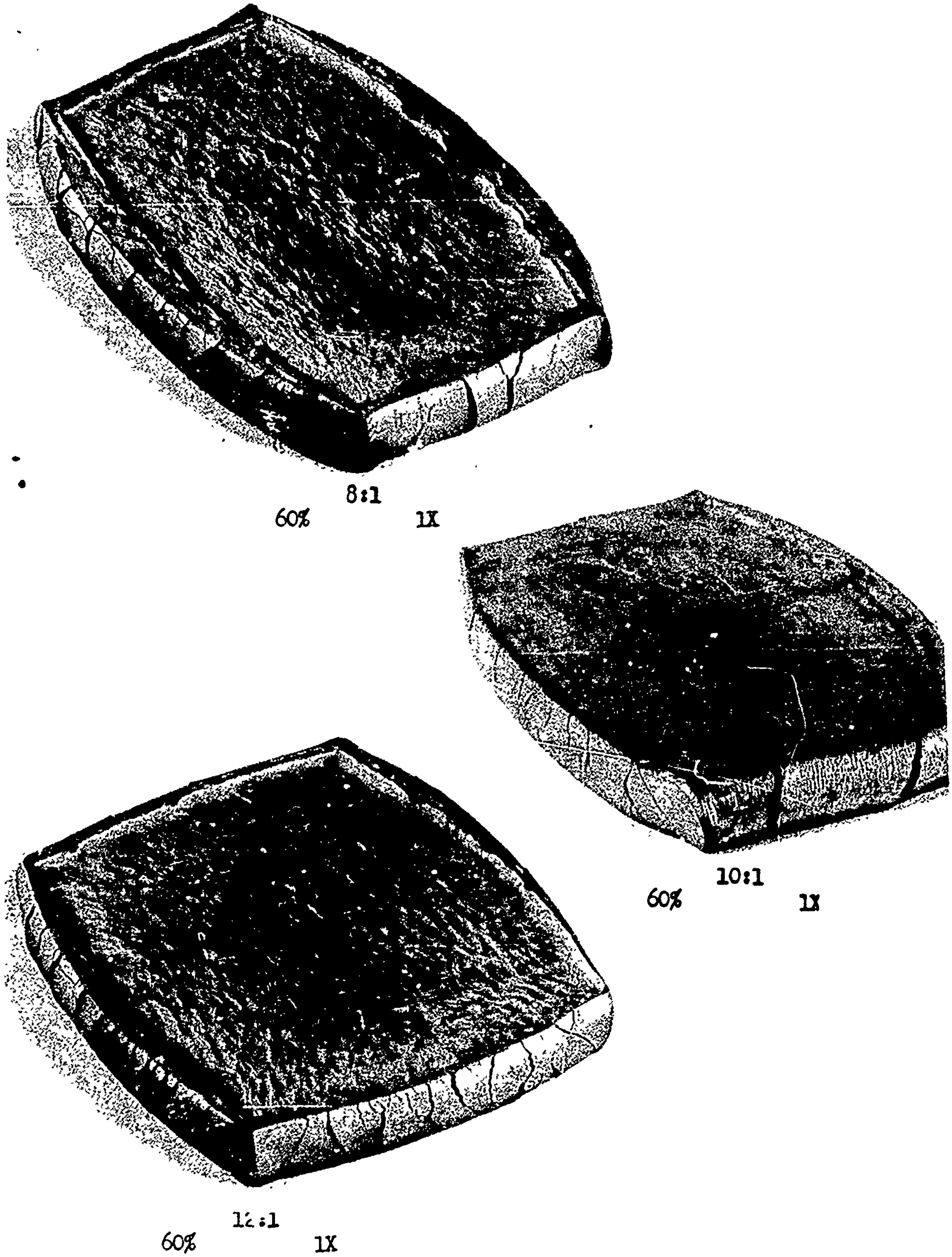
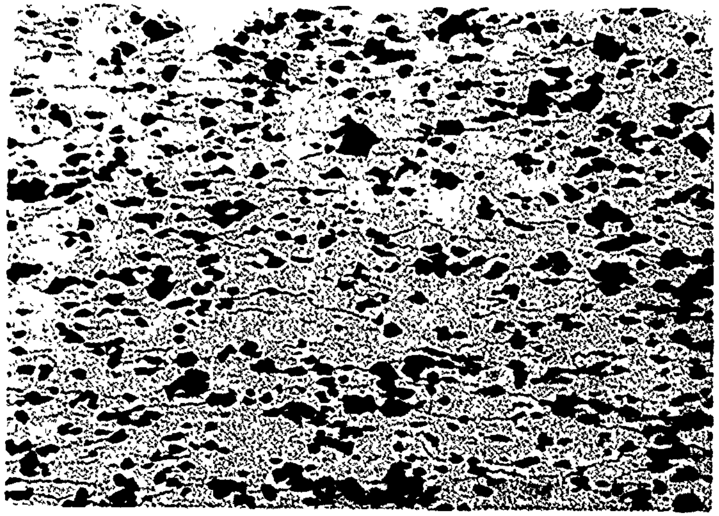
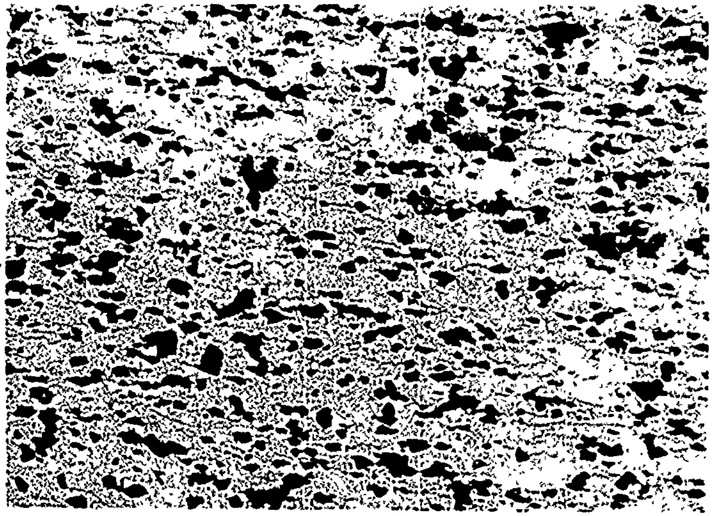


Figure 26 - Typical Samples Forged 60 Percent @2400 F



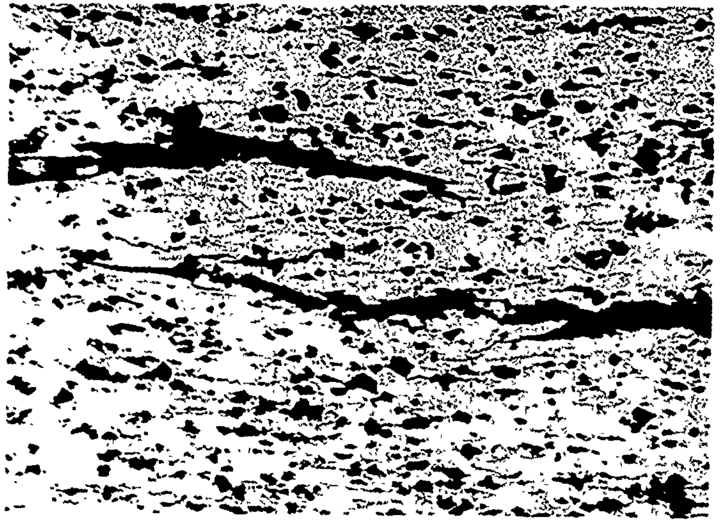
0-707 200X

a



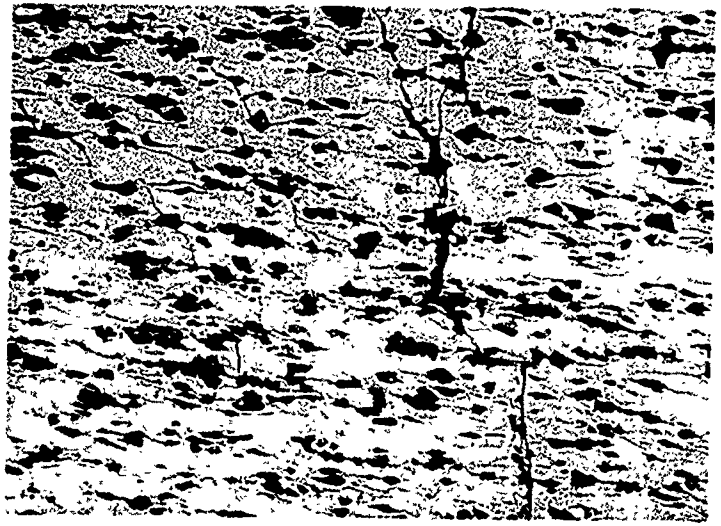
0-709 200X

b



0-708 200X

c



0-718 200X

d

Figure 27 - Forged Microstructures

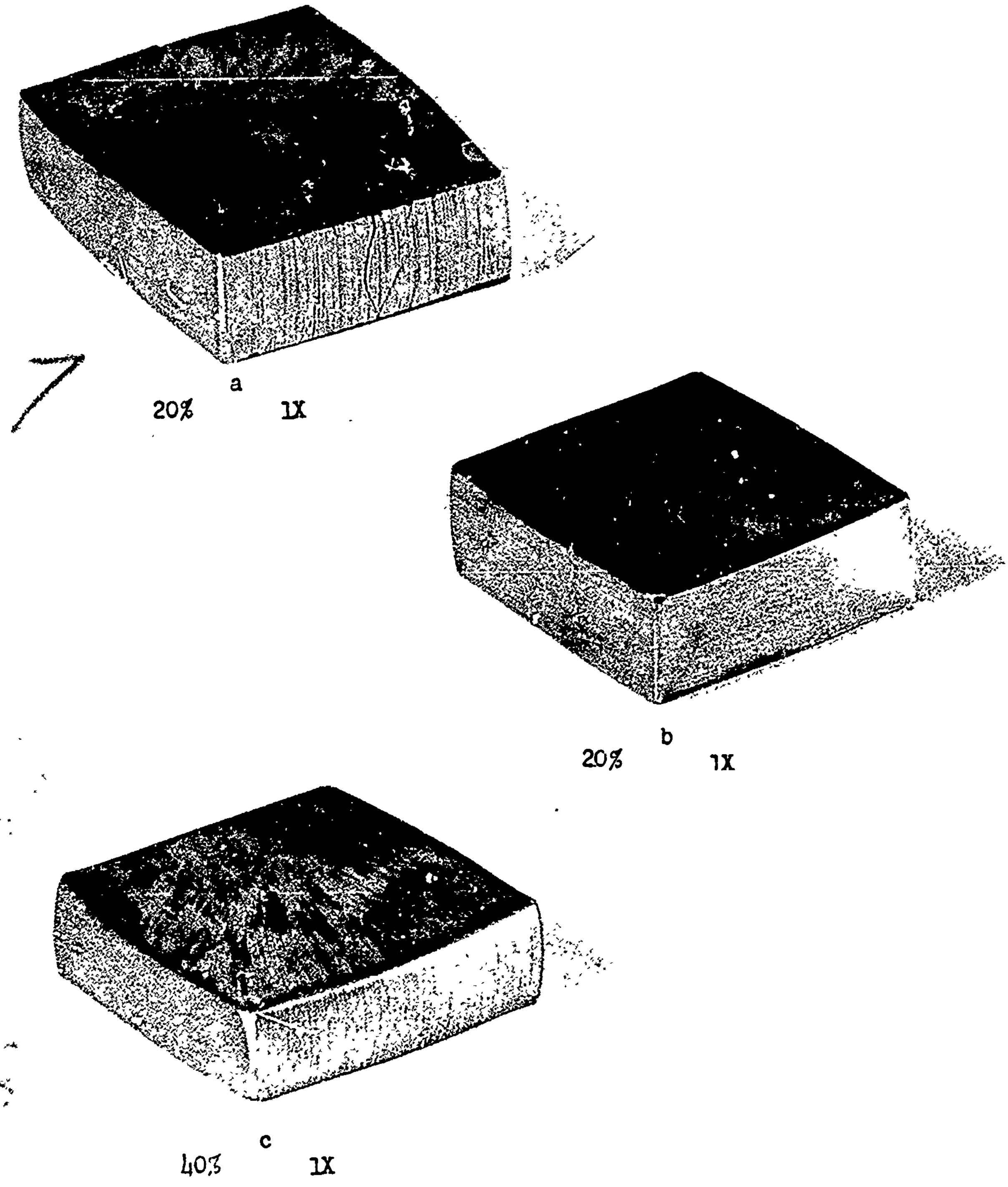
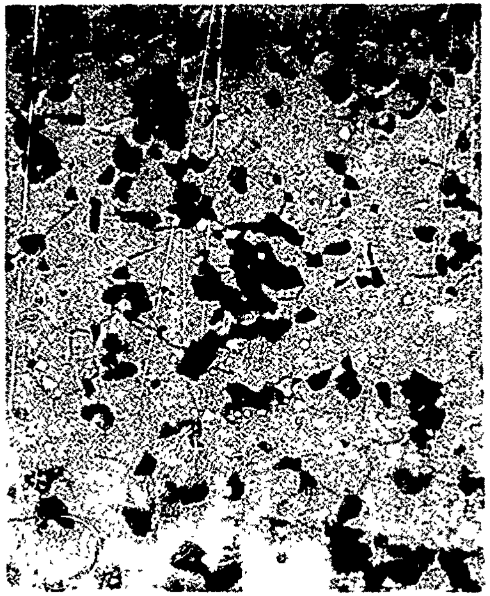
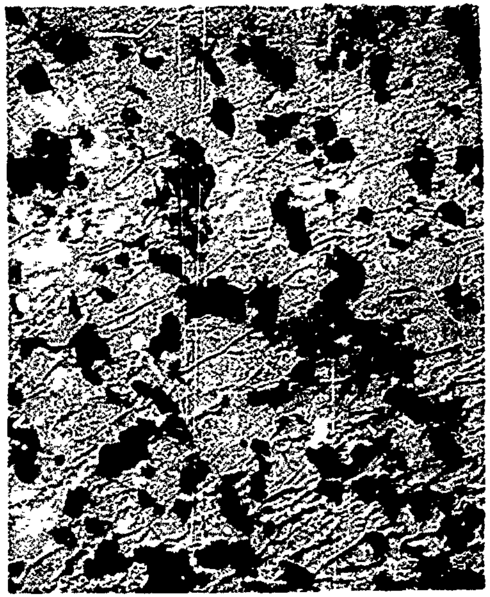


Figure 28 - Typical Samples Forged @400 F

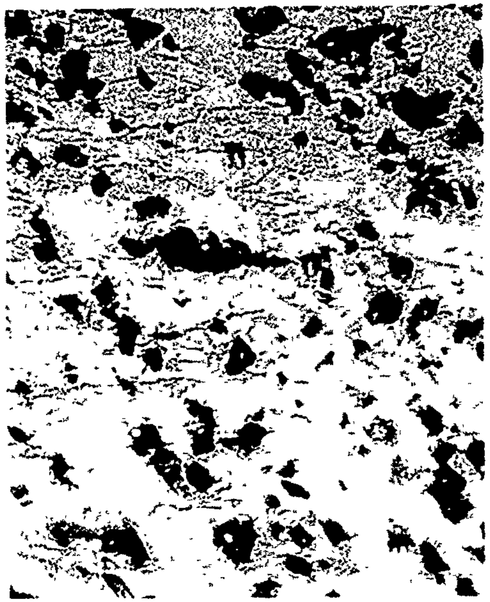


As Extruded



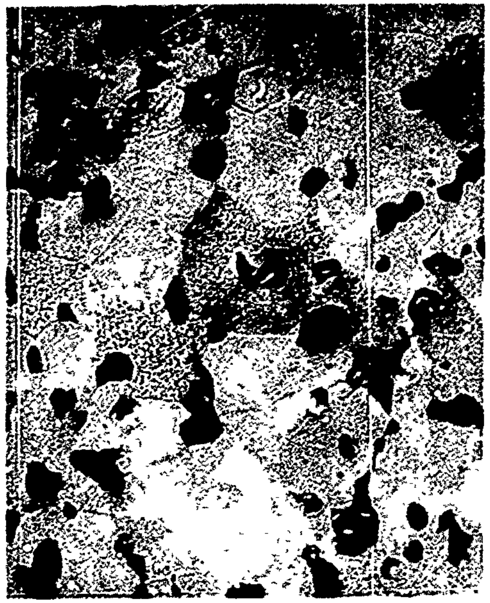
50% Reduction @500 F

Annealed $\frac{1}{2}$ Hr. @1000 F



Annealed $\frac{1}{2}$ Hr. @1600 F

Annealed $\frac{1}{2}$ Hr. @1800 F



Annealed $\frac{1}{2}$ Hr. @2900 F

Figure 29 - Recrystallization of Warm Forged Chrome-30 (Oblique Light; 480X)

annealing treatments at increasing temperature are shown in Figure 29. A corresponding softening curve, developed through microhardness measurements of the matrix grains, is shown in Figure 30. It can be seen from the microstructures that grain growth was retarded during the annealing treatment at 2900°F as a result of the dispersed MgO particles.

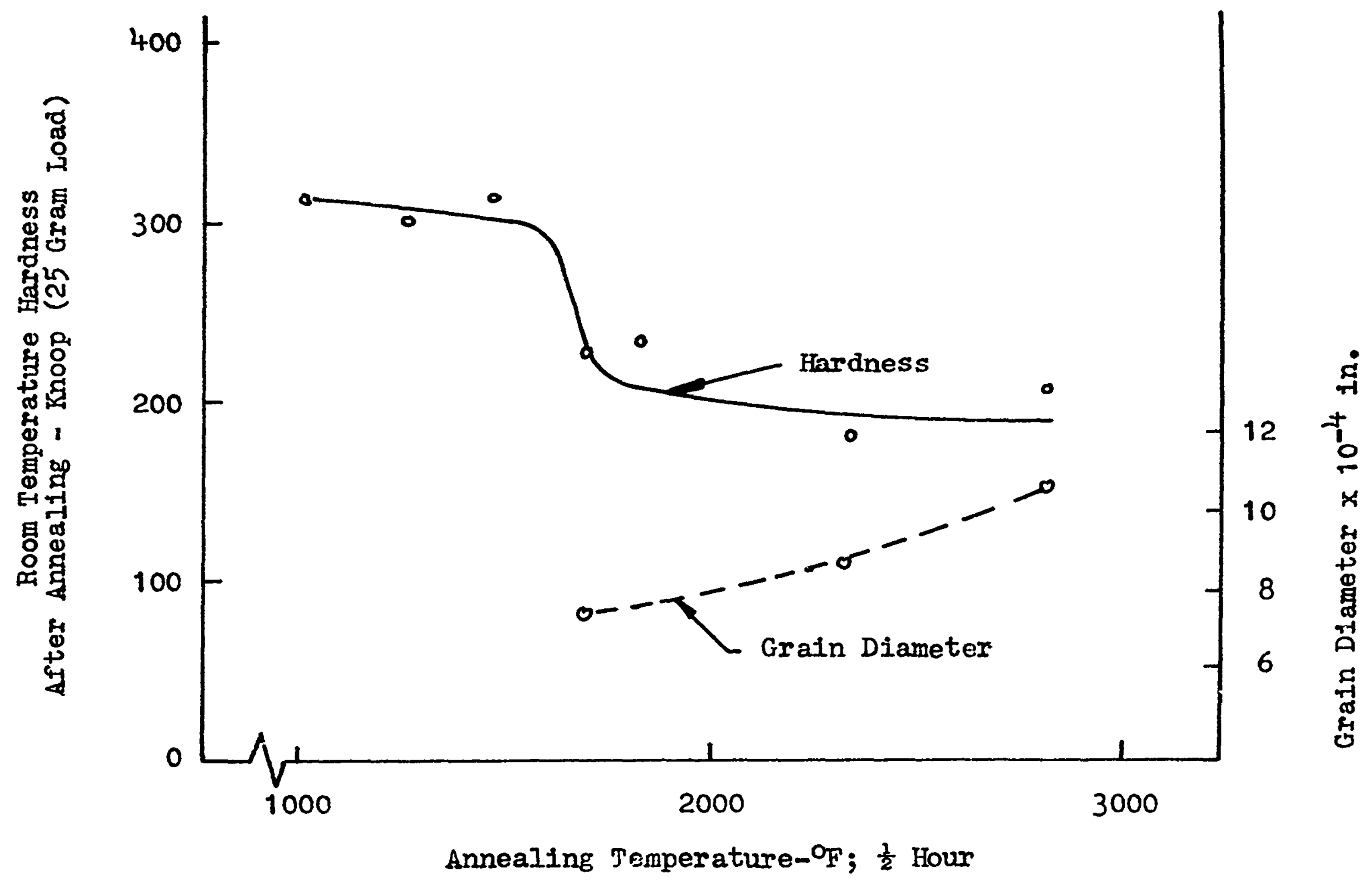


Figure 30 - Softening Behavior of Chrome-30 Forged 50 Percent @500 F

PRELIMINARY ROLLING STUDIES

Wedge Rolling Study

Twelve wedge-shaped samples were rolled to establish guide lines for the selection of principal rolling parameters. These rolling trials were designed to investigate: (1) rolling temperatures from 400 to 2200° F; (2) rolling direction; (3) heating atmosphere; (4) surface protection; and (5) reduction per pass. The wedge samples were designed to cover a range of rolling reductions from 0 to 75 percent when rolled to a thickness of 0.100 inches. Wedge designs for both parallel and transverse rolling are illustrated in Figure 31, and a summary of the rolling trials is given in Table 4.

A 15 mil electroplated nickel cladding was applied to four of the wedges scheduled to be rolled at 1800, 2000 and 2200° F. These coatings separated from the wedges, however, during the first roll pass; and excessive surface cracking occurred, as it did for wedges which were heated in air and rolled unprotected. Heating in argon offered no improvement. Wedges rolled at temperatures from 400 to 1200° F were free of surface cracks but experienced severe edge splits when reduced beyond 60 percent. Reductions per pass of approximately 10 percent and 20 percent appeared comparable and little difference was noted in the rollability of parallel and transverse wedge sections. The rolled wedge samples are shown in Figures 32 through 36.

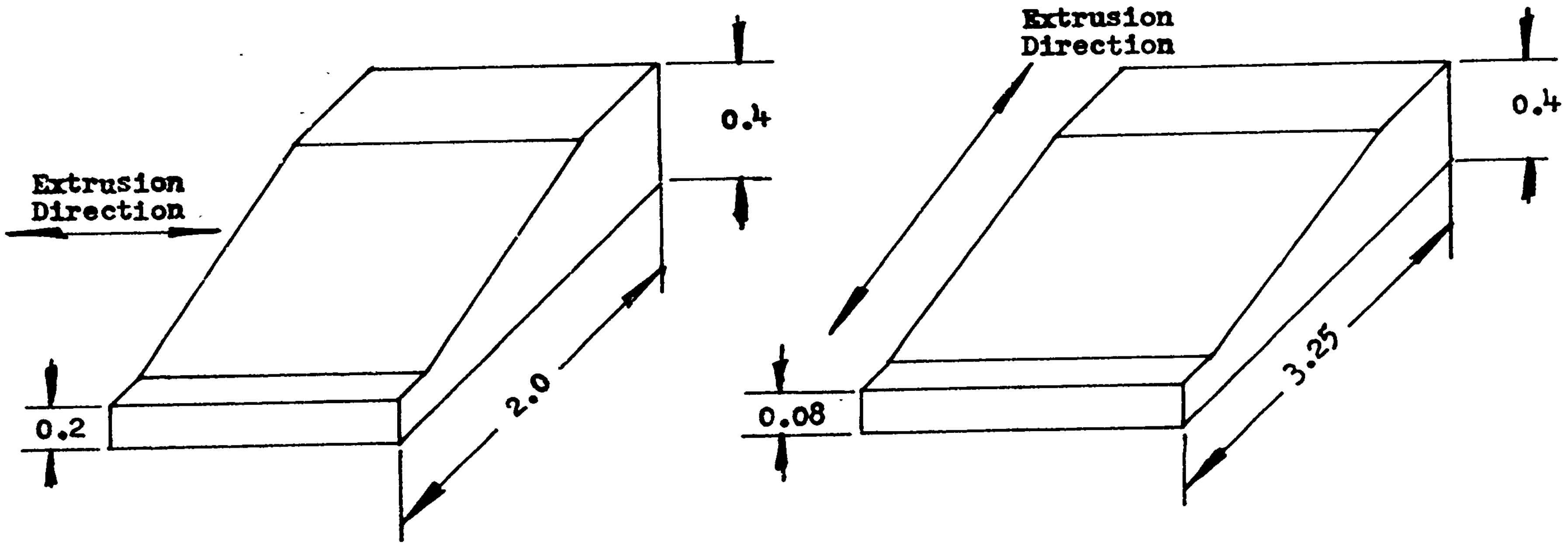
The hardness data presented in Table 5 indicate that considerable work hardening occurred at all rolling temperatures, including 2200° F.

It can be seen, from Figure 37, that excessive hardening developed in wedge locations which had reduction as low as 15 percent. These data show, as expected, very little change in hardness with increasing reduction for rolling temperatures of 1800° F and above. Hardness levels increased proportionately with increased reduction when wedges were rolled below the recrystallization temperature. Vickers hardness values obtained after annealing at 1800° F for one half hour were consistent within a 30 point scatter and independent of rolling temperature. It was apparent, however, that the average hardness level after annealing was 10 to 20 points above the hardness level of extruded material (indicating insufficient annealing temperature or time).

It was concluded, as a result of this study, that a suitable cladding technique should be developed in order to successfully hot roll chromium composite sheet bars. It was also established that: (1) hot rolling temperatures of 1800° F and above should be employed; (2) warm rolling temperatures of 400 to 1200° F could be used to provide 50 to 60 percent total reduction; and (3) reductions per pass of 10 to 20 percent or greater could be successfully used at both hot and warm rolling temperatures.

Preliminary Warm Rolling Breakdown Trials

A total of twelve sheet bars were rolled without cladding to evaluate rollability in the



Transverse to Extrusion Direction

Parallel to Extrusion Direction

Figure 31 - Configuration of Wedge Rolling Samples

Table 4. Summary of Wedge Rolling Trials

Wedge Number	Surface Clad	Rolling Temperature, F	Rolling Direction(a)	Average Reduction Per Pass, Percent	Number of Passes	Maximum Reduction, Percent	Remarks
3	None	400	P	9.7	10	64.9	Crack on Hi red. end at 47.8%. Sample broke.
2	None	600	P	10.5	12	73.8	1½" split on Hi red. end.
1	None	800	P	8.9	12	67.7	Sample split on flattening.
4a	None	800	T	10.3	8	58.2	Slight cracks on Hi red. end at 54%.
11a	None	800	P	16.5	7	71.7	Hi red. end cracks at 62.3%. Slight edge cracks.
10a	None	1200	P	18.6	6	71.4	Two side splits parallel to edges on last pass.
5	Nickel	1800	P	20.4	2	36.6	Plating off after 1st pass. Sample split both sides.
12a	None	1800(b)	P	9.8	11	68.0	Large split - trailing end.
6	Nickel	2000	P	22.1	4	64.5	Plating off after 1st pass, severe surface cracks.
9	Nickel	2200	T	10.1	5	41.4	Plating off after 1st pass, severe surface cracks.
7	None	2200	P	21.1	5	69.5	Severe surface cracks.
8a	Nickel	2200	T	19.9	3	48.7	Severe surface cracks. No edge cracks.

(a) P - Rolled parallel to extrusion direction; T - Transverse to extrusion direction.

(b) Heated in Argon. All others heated in Air.

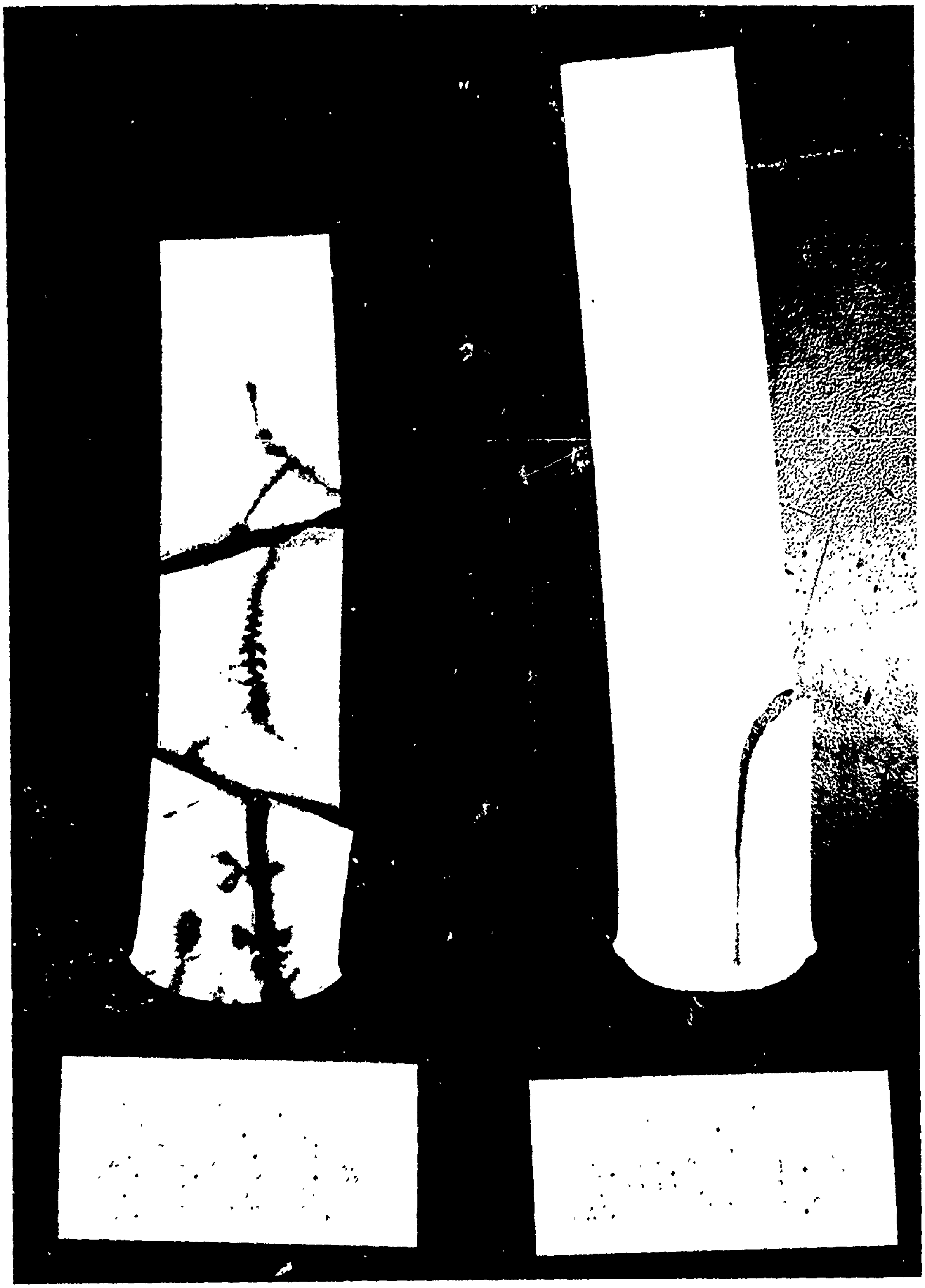


Figure 32 - Rolled Wedge Samples

Figure 33 - Rolled Wedge Samples

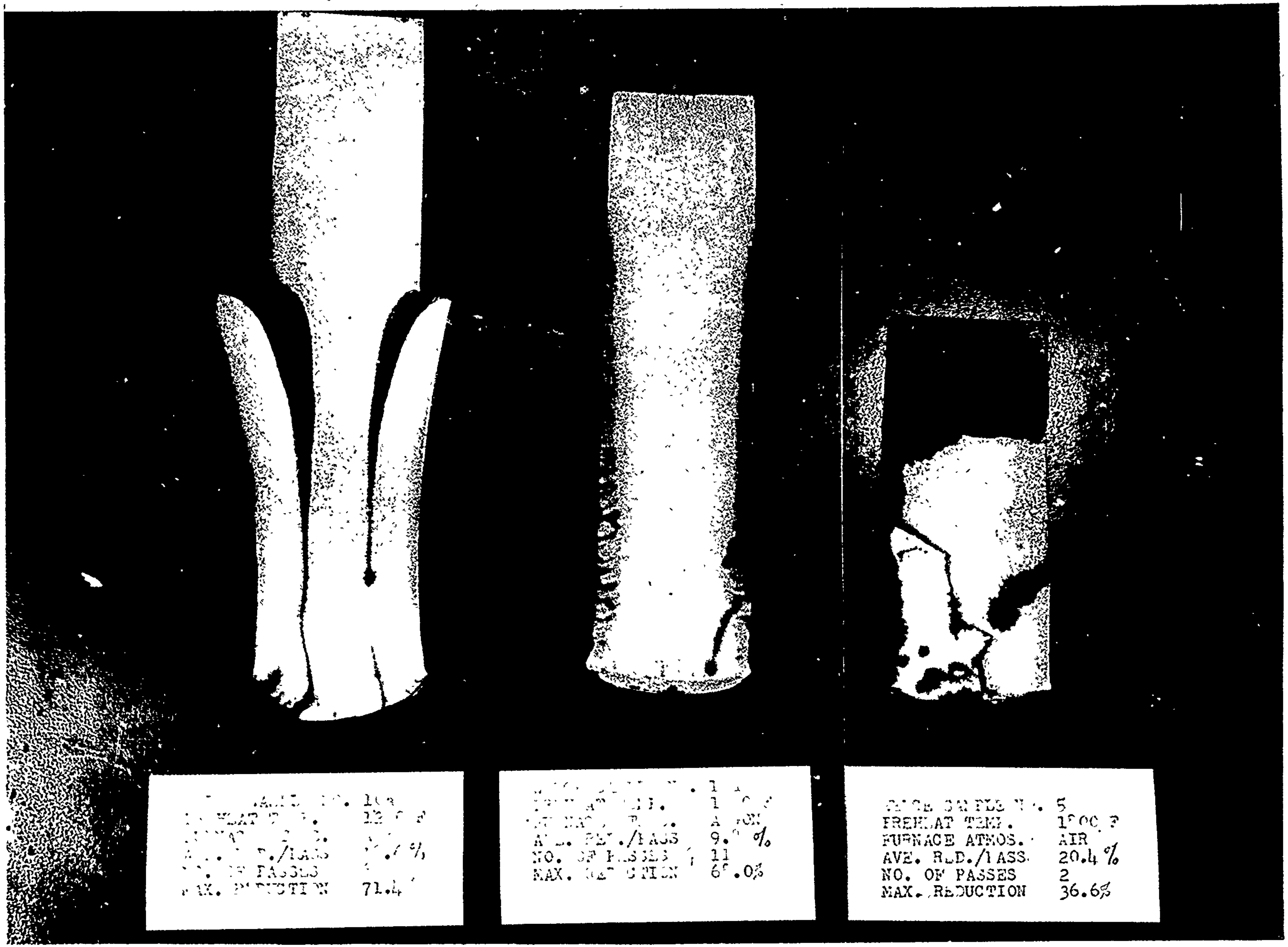


Figure 34 - Rolled Wedge Samples

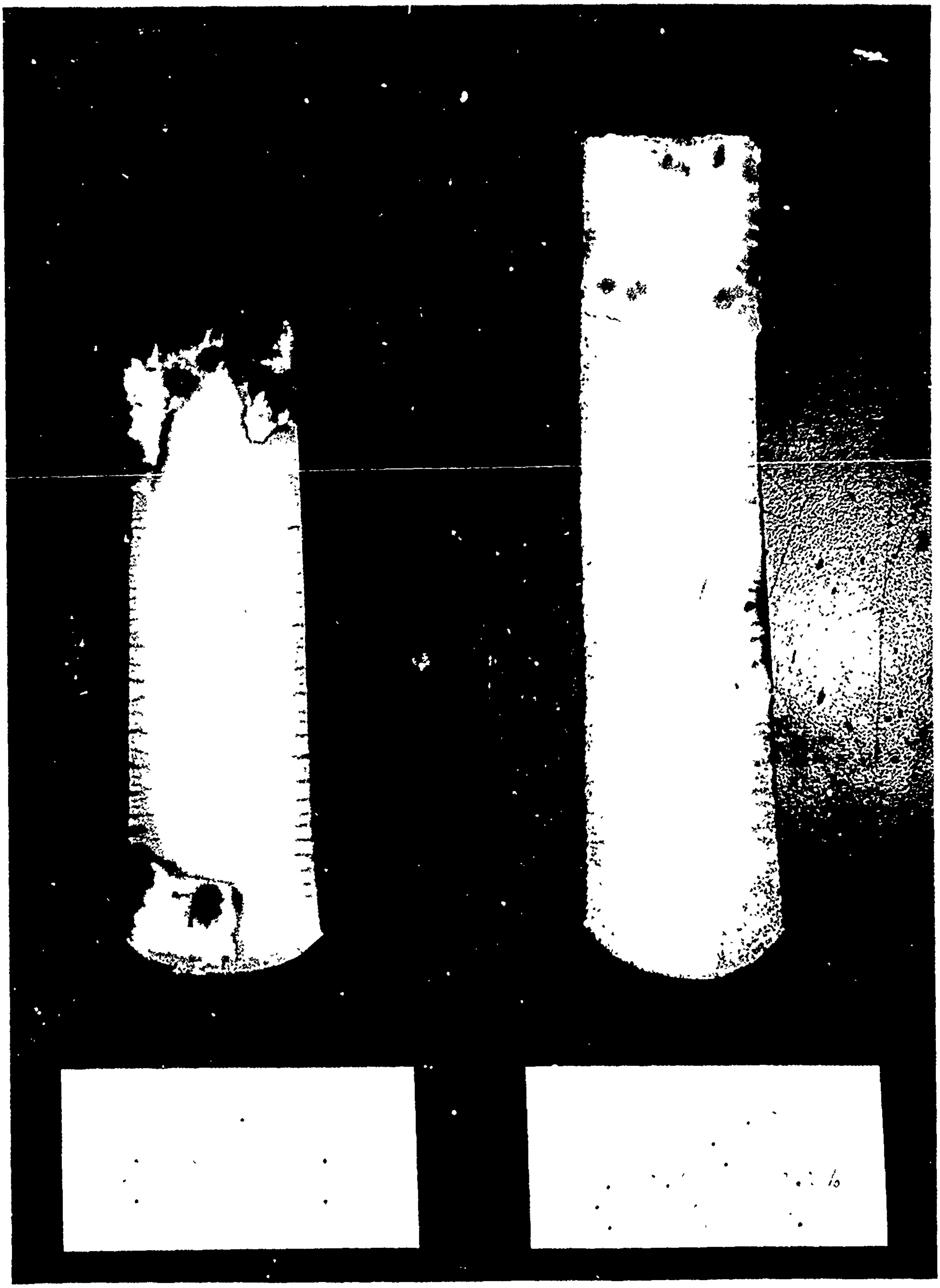


Figure 35 - Rolled Wedge Samples

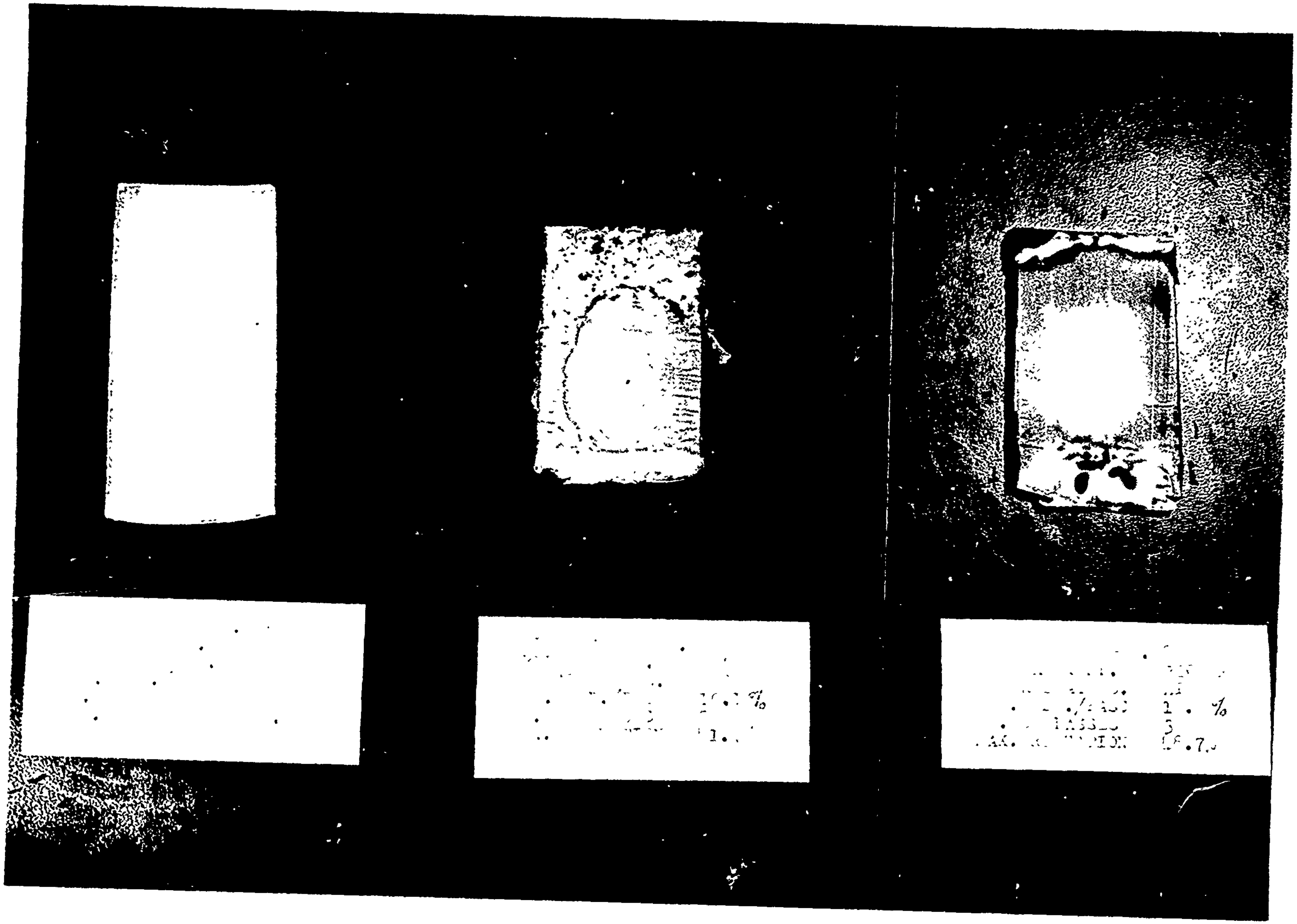


Figure 36 - Rolled Wedge Samples

Table 5. Effect of Rolling Temperature and Reduction on the as Rolled and Annealed Hardness of Wedge Samples

Wedge Sample Number	Rolling Temperature, F	Vickers Hardness, 10 Gram Load			
		15 Percent Reduction	30 Percent Reduction	45 Percent Reduction	60 Percent Reduction
		As Rolled			
3	400	209	222	242	267
2	600	240	256	266	281
1	800	221	254	274	289
4	800	222	228	243	260
11	800	236	247	258	274
10	1200	238	264	272	285
5	1800	232	235	-	-
12	1800	249	253	254	253
6	2000	210	224	225	225
9	2200	182	176	176	-
7	2200	210	213	210	215
8	2200	198	197	199	-
		Hydrogen Annealed ½ Hour at 1800°F			
3	400	162	153	151	157
2	600	173	168	171	169
1	800	153	154	164	168
4	800	150	150	151	160
11	800	165	161	166	176
10	1200	168	167	165	170
5	1800	173	157	-	-
12	1800	159	159	158	158
6	2000	159	159	164	161
9	2200	175	168	168	-
7	2200	161	166	169	181
8	2200	175	177	144	-

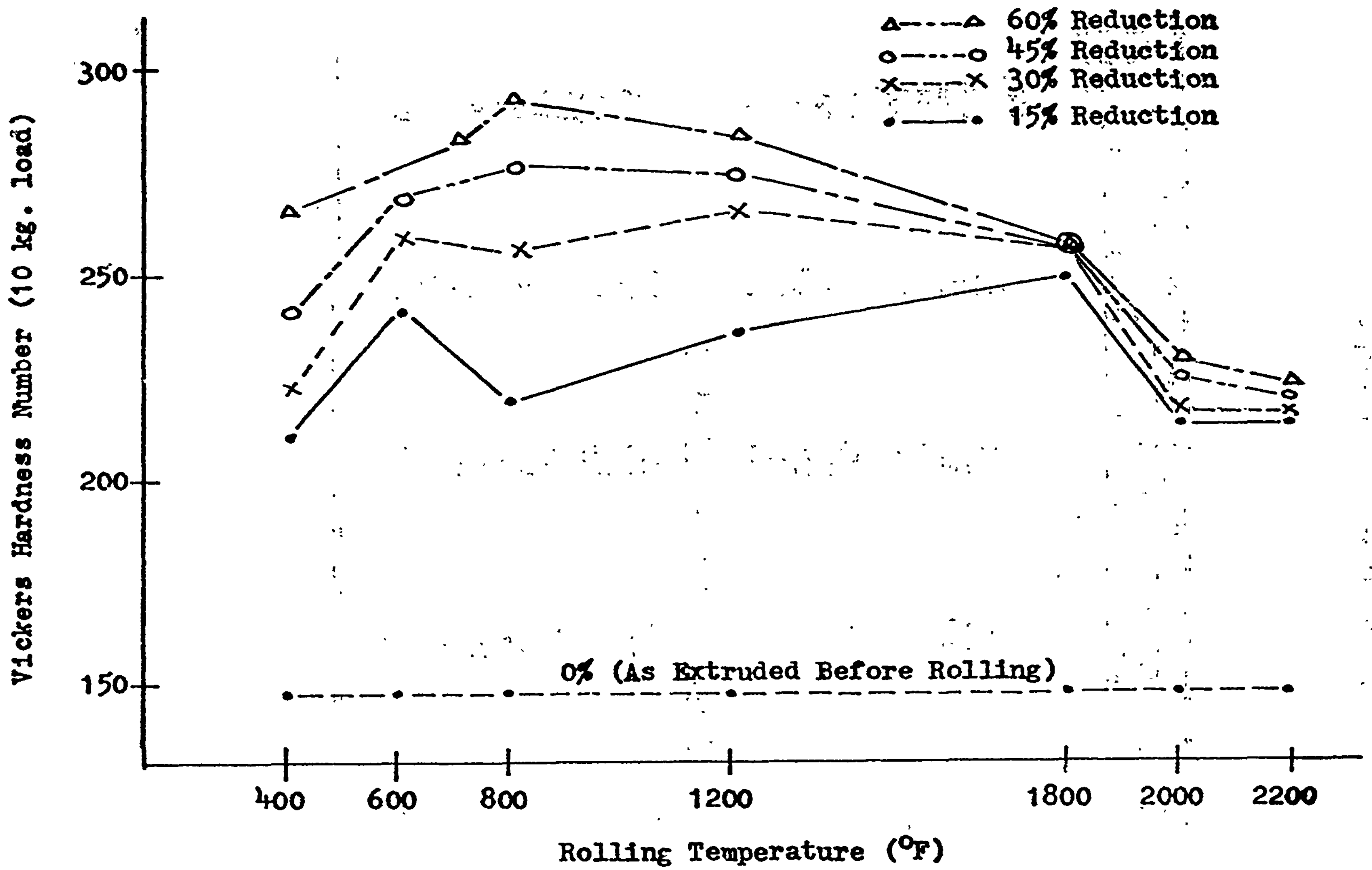


Figure 37 - Effect of Rolling Temperature and Reduction on the As-Rolled Hardness of Wedge Samples

800 to 1200°F temperature range. Additional variables of rolling direction, annealing temperature and annealing atmosphere were evaluated. The details of these trials are summarized in Table 20 in the Appendix.

It was discovered that chromium composite sheet bars could be warm rolled only in the range of 40 to 56 percent reduction at 800°F before annealing was required to soften the severely worked structure and prevent propagation of edge splits. Rollability was also found to be considerably reduced in all cases after intervening one half hour anneals at 1800°F. Work hardening and response to annealing for various reductions are indicated by the hardness data tabulated in Table 6. Hardness values ranging from 78 to 84 Rockwell B, indicate that the annealing temperature and time were sufficient to adequately soften sheet surfaces. Apparently the inability to obtain similar reductions after annealing was caused by a pick-up of impurities from the furnace atmosphere or an unequal stress distribution accompanying retained internal stresses and roll deflection. Increasing the annealing temperature to 2000°F was of no benefit. Rolling at 1200°F and annealing at 2000°F provided no improvement. Rolled sheet bars 14, 15 and 21, shown in Figures 38, 39 and 40 respectively, illustrate this warm rolling breakdown problem. Rolled sheet bars 16 and 24, shown in Figures 41 and 42 respectively, were rolled parallel to the extrusion direction. Bar 16 split on the third roll pass while bar 24 fractured during the second pass.

In view of realistic economics it was concluded that a warm rolling breakdown procedure would be impractical and that conventional hot-warm rolling should be employed.

Evaluation of Cladding Techniques

It was discovered during the wedge rolling trials that unprotected chromium composite could not be satisfactorily hot rolled. This finding was verified by the rolling of a completely machined sheet bar (#22), shown in Figure 43, which was heated in argon to 2200°F prior to each of four rolling passes. It was subsequently learned that the flame sprayed nickel cladding, applied to sintered billets prior to extrusion, offered inadequate protection for hot rolling. The sheet samples shown in Figures 44 and 45 were heated in air and argon respectively to 2200°F prior to each of six rolling passes. Although the sprayed nickel cladding remained intact, splits occurred at the unprotected sheet edges and on surfaces underlying defects in the 10 mil clad. Micro-Kjeldahl analyses performed on specimens of sheet 29 (Figure 43) revealed the following nitrogen contents at the contaminated edge and center sections underlying the nickel cladding:

Sample	Sample Location	Nitrogen Content PPM
Sheet 29	Unprotected Edges	950
Sheet 29	Center of Sheet	430
As extruded (861)	Center of Sheet Bar	80

Because these results for sheet 29 are average core-surface values, the actual nitrogen contamination within outer surface layers would be considerably higher. The normal nitrogen content for starting sheet bars was shown to be 80 PPM.

The following (4) four techniques were evaluated in an attempt to provide improved sheet bar protection for hot rolling: (1) 40 mil flame spray nickel coating applied over

Table 6. Effect of Annealing Temperature on the Reduction and Hardness of Warn Rolled Sheet Bars

Trial No.	Annealing Temperature, F[a]	First Reduction, Percent	Second Reduction, Percent	Third Reduction, Percent	Rockwell B Hardness					
					After First Reduction	After First Anneal	After Second Reduction	After Second Anneal	After Third Reduction	After Third Anneal
13	1400 Argon	50.8	-	-	95	95	-	-	-	-
14	1800 Argon	47.5	43.8	24.0	94	87	100	84	103	84
17	1800 Argon	50.4	37.4	-	97	79	100	-	-	-
19	1800 Argon	40.3	24.8	-	93	78	98	82	-	-
21	1800 Argon	56.2	35.4	-	101	81	102	-	-	-
23	1800 Argon	40.5	22.3	24.6	94	79	95	81	99	-
15	1800 H_2	47	31.0	-	97	80	99	83	-	-
25	2000 Argon	41.5	29.0	29.5	93	79	97	82	101	84
26[b]	2000 Argon	38.4	30.3	20.9	95	79	102	83	100	81

(a) One half hour at temperature.

(b) Rolled at 1200°F. All others rolled at 800°F.

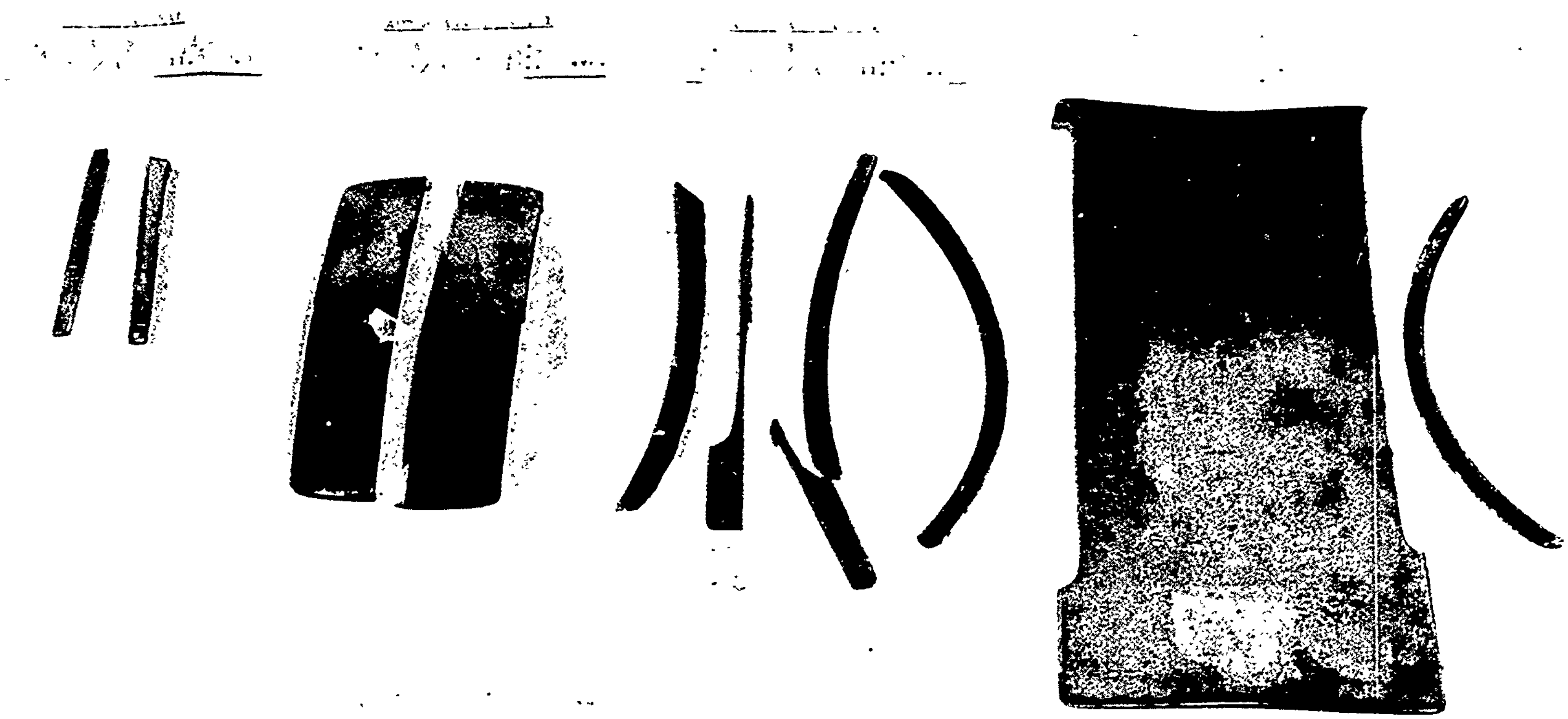


Figure 38 - Warm Rolled Sheet #14

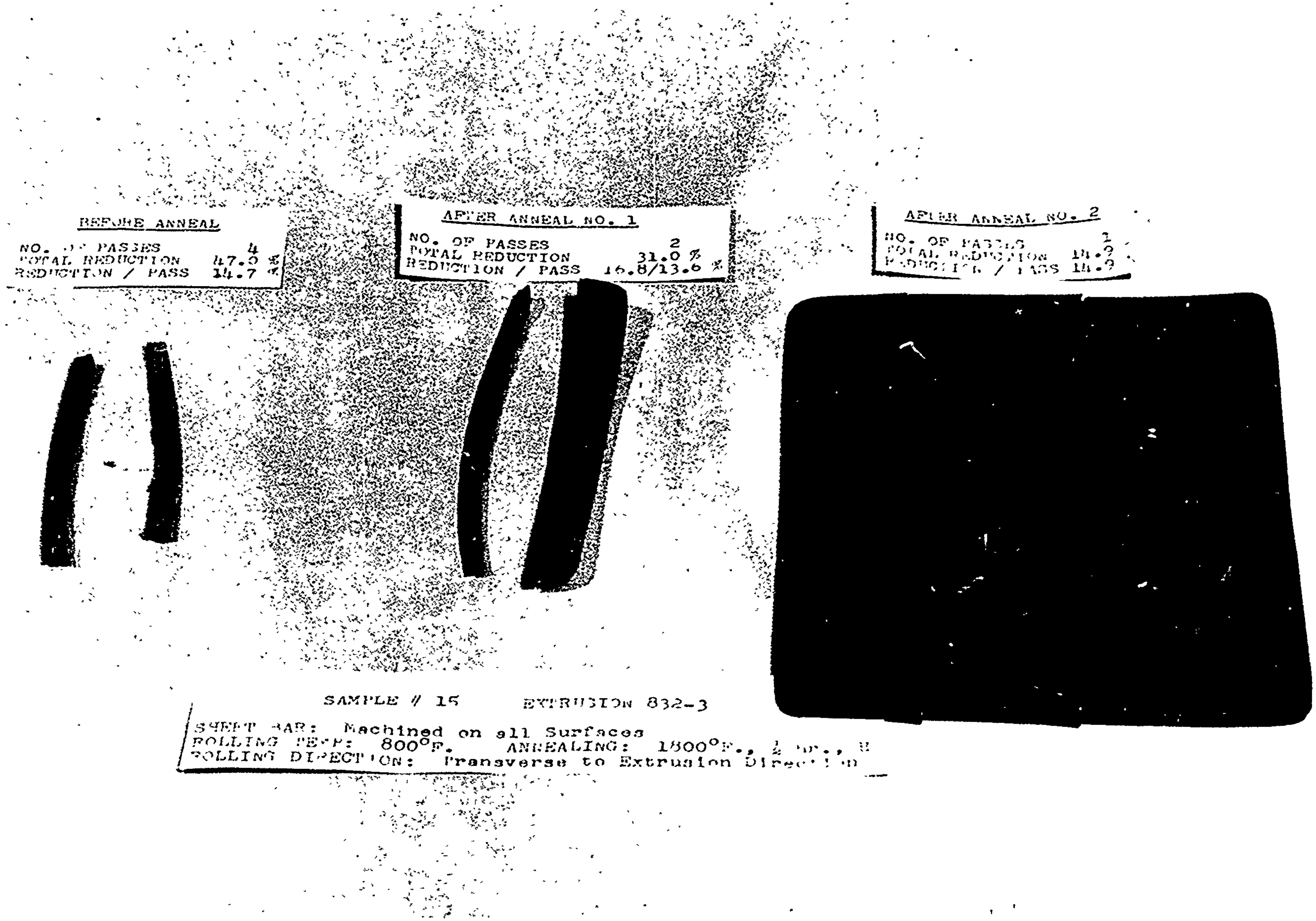


Figure 39 - Warm Rolled Sheet #15

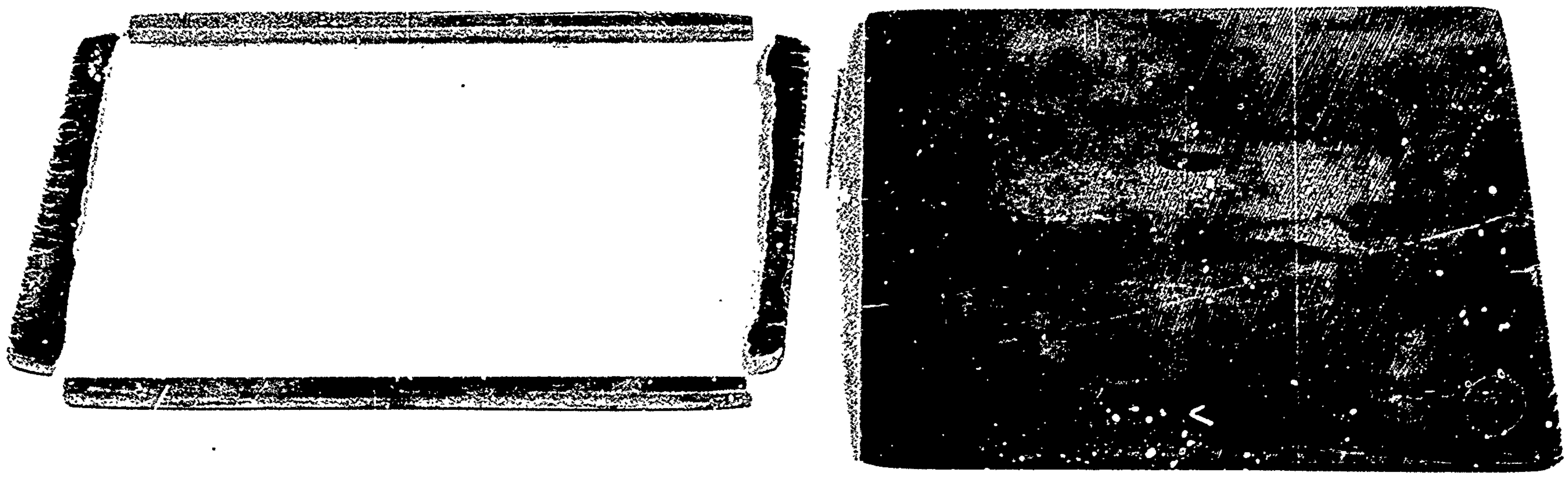


Figure 40 - Warm Rolled Sheet #21

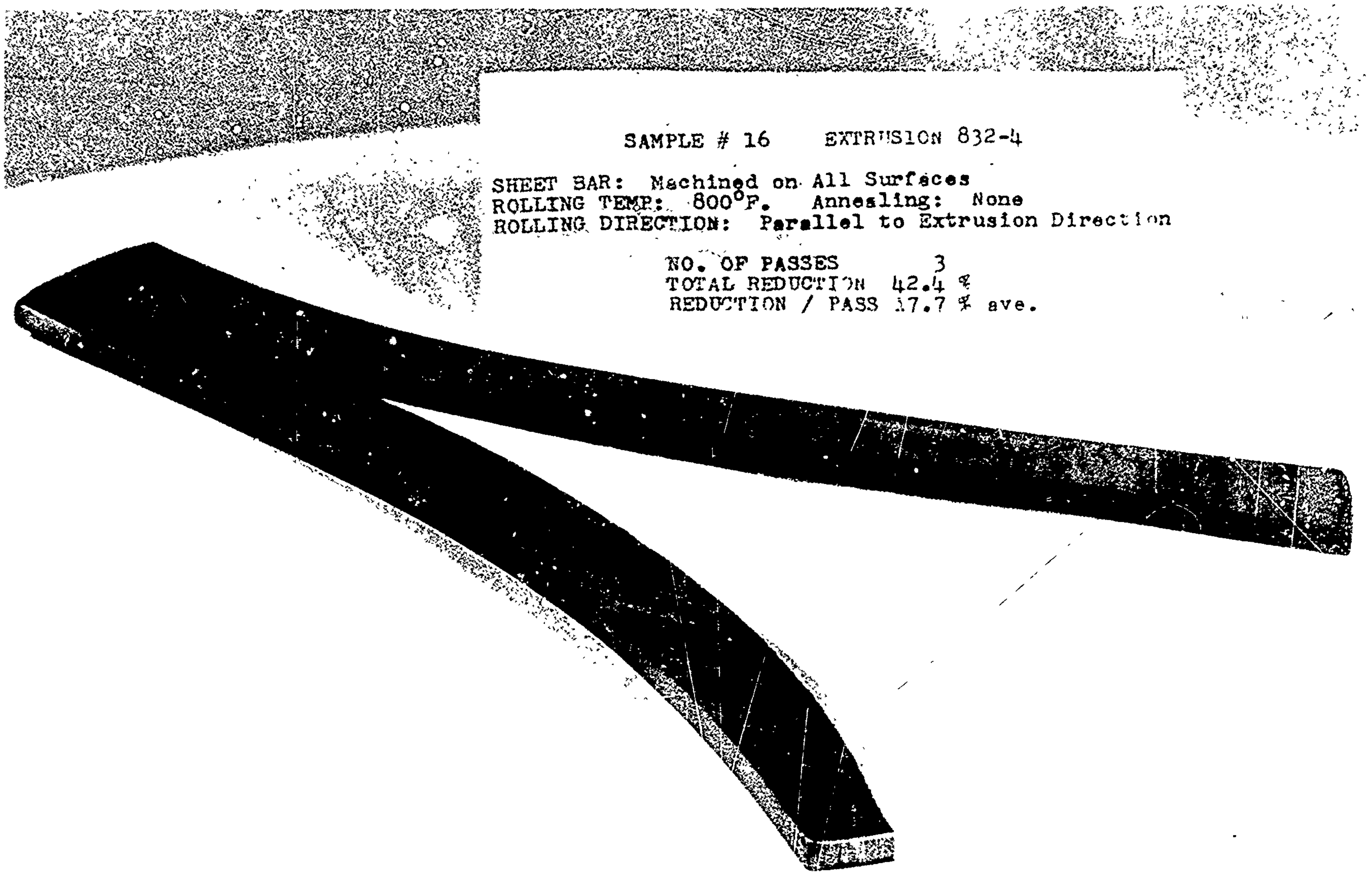


Figure 41 - Warm Rolled Sheet #16

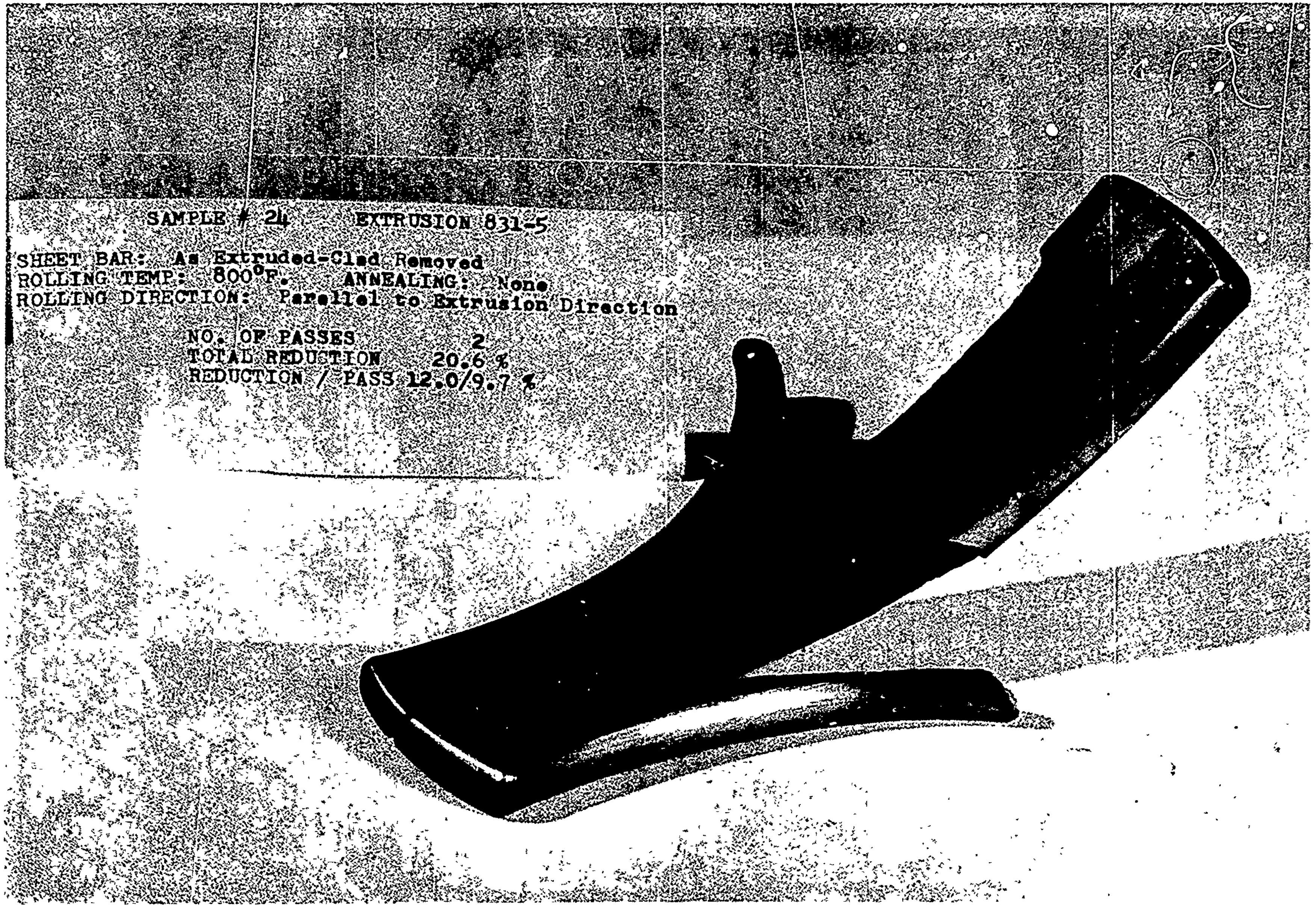


Figure 42 - Warm Rolled Sheet #24

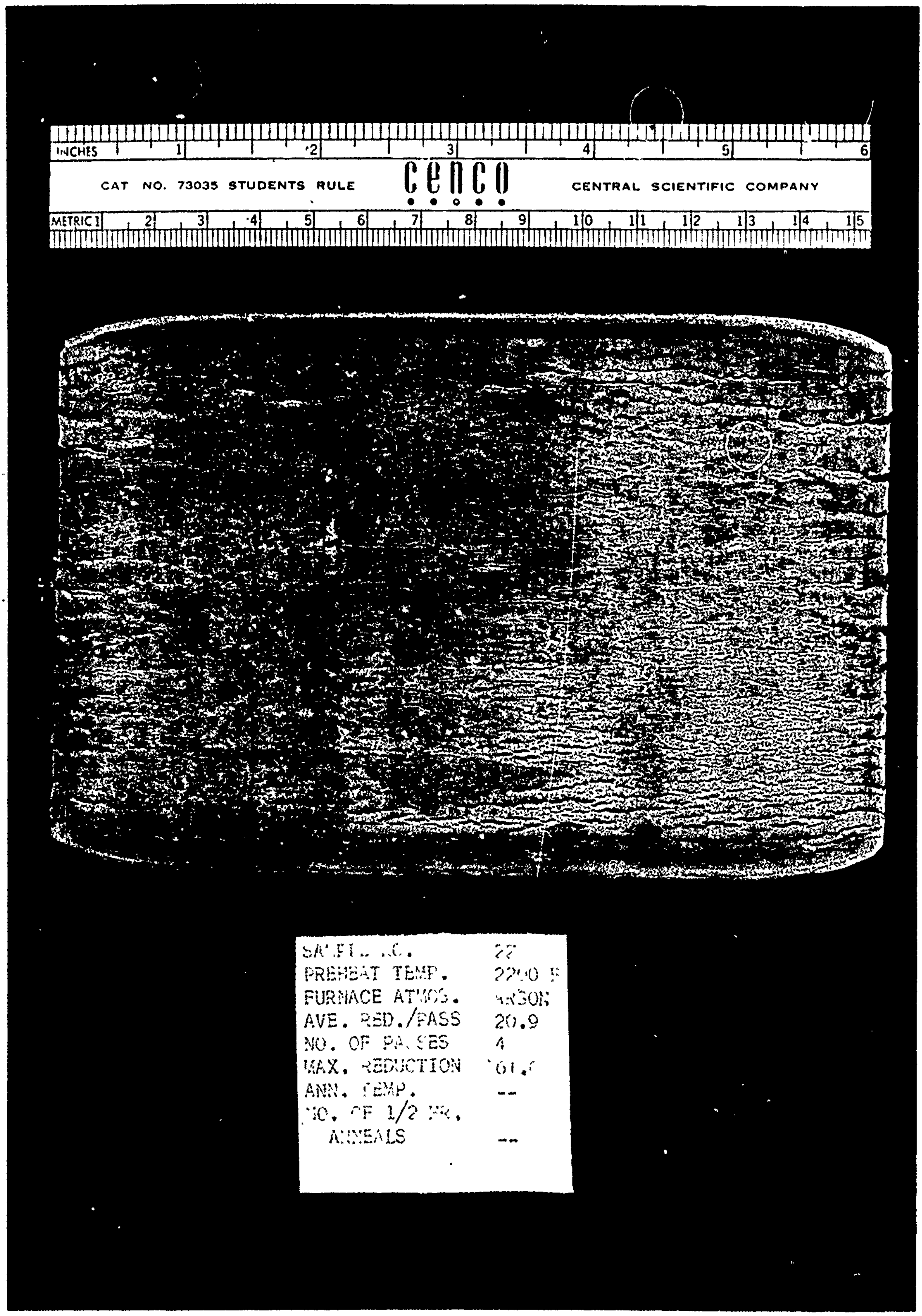


Figure 43 - Hot Rolled Sheet #22

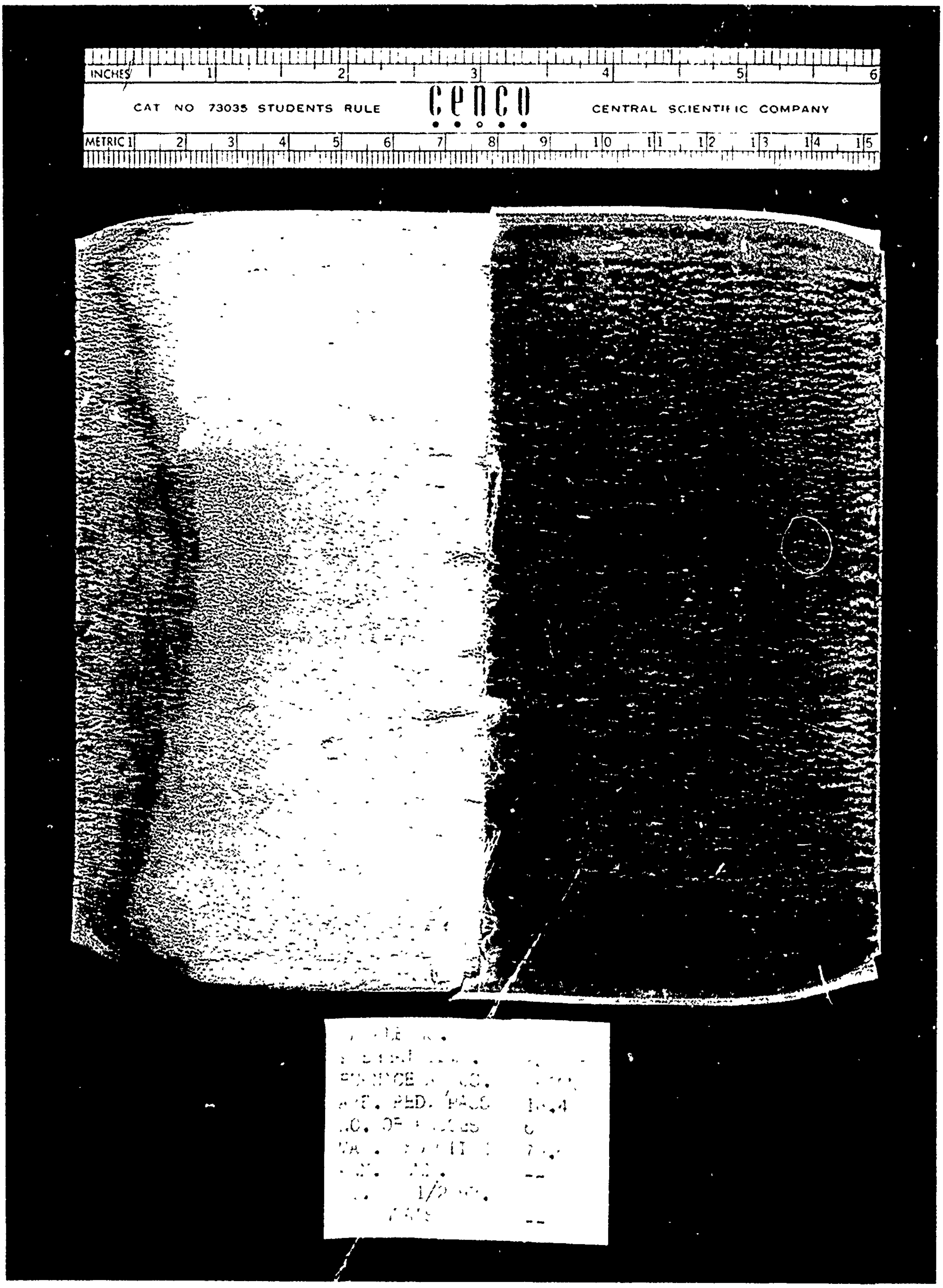


Figure 44 - Hot Rolled Sheet #29

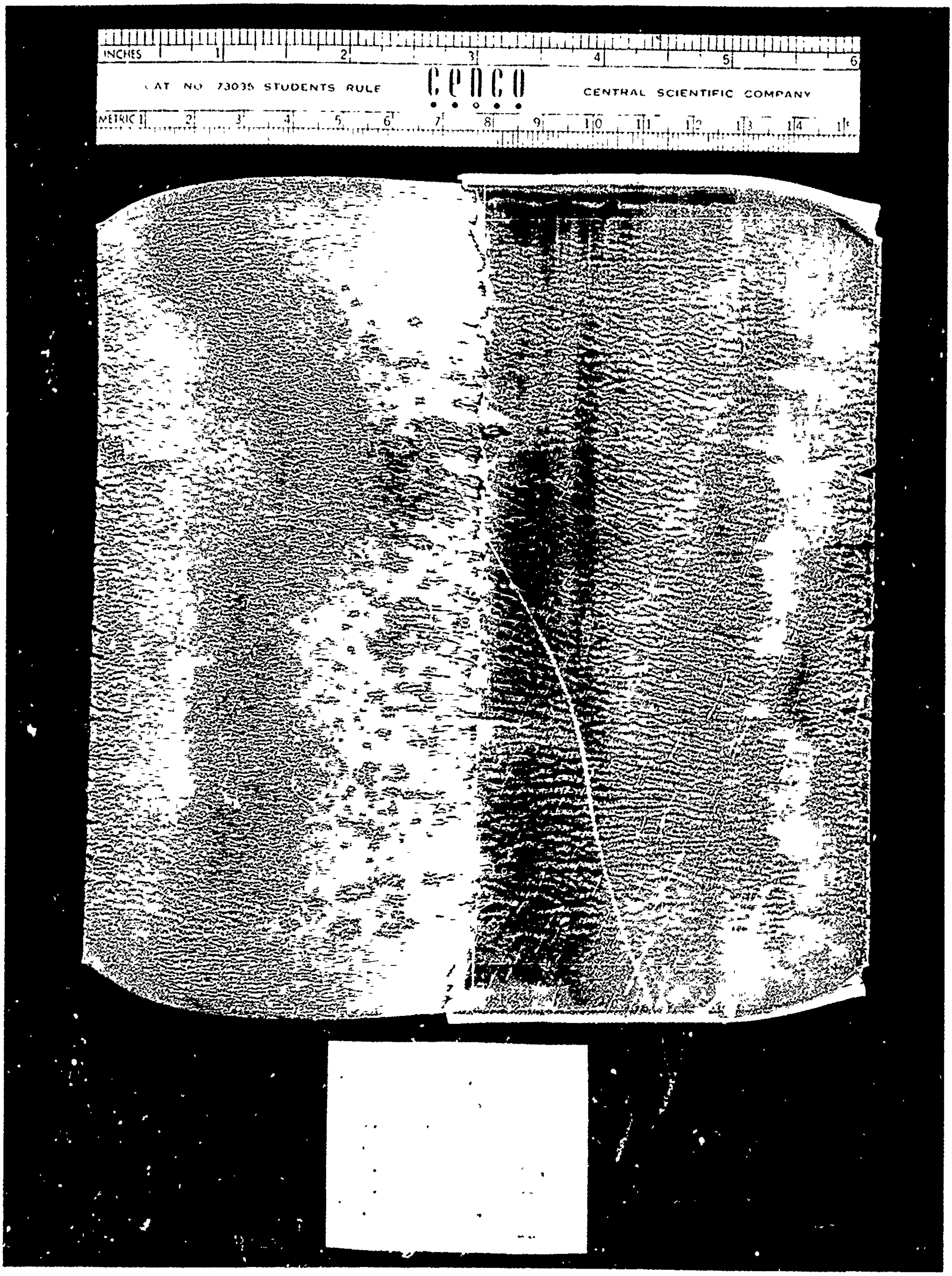


Figure 45 - Hot Rolled Sheet #30

the extruded cladding; (2) 15 mil electroplated nickel coating; (3) salt bath heating, and (4) steel "picture frame" enclosure. These rolling trials and those mentioned above are summarized in Table 21 in the Appendix.

Additional Flame Sprayed Nickel

Extruded sheet bar 35, containing the original nickel cladding, was coated on all surfaces with an additional 40 mil layer of nickel using standard wire metalizing techniques. This sheet sample, shown in Figure 46, was heated to 2200° F in a hydrogen atmosphere prior to each of (10) ten roll passes. A significant improvement in surface condition was noted, but coating separation on the ends of the sheet bar exposed unprotected material and resulted in typical edge splits.

Nickel Electroplate

Sheet samples 32 and 33, shown in Figures 47 and 48, were heated in argon and hydrogen respectively to 2200° F prior to each of (9) nine roll passes. These sheet bars were prepared for rolling by machining to remove the extruded nickel cladding and subsequent treatment in a nickel plating bath to provide a 15 mil deposit. The plated samples were heated in a hydrogen atmosphere for 2 hours at 2250° F to provide a nickel rich diffusion layer for added protection during rolling. Although the plating was badly bubbled and contained excessive porosity, the rolling proceeded satisfactorily and produced a tightly adherent cladding on both samples. Splits originated, however, from exposed ends where the plating separated during rolling, and surfaces beneath the clad, although free of cracks, were badly wrinkled.

Salt Bath Heating

Sheet bar #36, with the extruded nickel clad intact, was sectioned into three 2 inch lengths which were heated to 2100° F in a $BaCl_2$-NaCl salt bath and rolled as indicated in Figure 49. The rolled samples showed evidence of reaction with the salt on unprotected edges and extensive edge splitting occurred.

Picture Frame Assembly

The rolled sheet shown in Figure 50 was rolled at 2200° F from a sheet bar containing between 1/8 inch thick steel cover plates welded to a 1 inch steel frame. This technique proved to be highly successful in providing hot rolled sheet free of edge and surface defects. Hardness measurements taken on as-rolled samples of this sheet indicated that considerable work hardening had occurred during the last roll pass. The sheet also displayed an apparent brittleness as evidenced by corner and edge breaks which occurred upon removal of the sheet from the frame. Spectrographic analysis of this sheet revealed a high iron concentration, which was found to be confined within a 2 mil surface layer. A hot hydrochloric acid pickle was successfully used to remove this contaminated material.

At the conclusion of this investigation, it was decided that the framing technique would be used to protect the chromium composite sheet bars during all hot rolling experiments. It was also decided that all framed sheet would be annealed for 1/2 hour after the last roll pass and pickled in hot concentrated hydrochloric acid after frame removal.

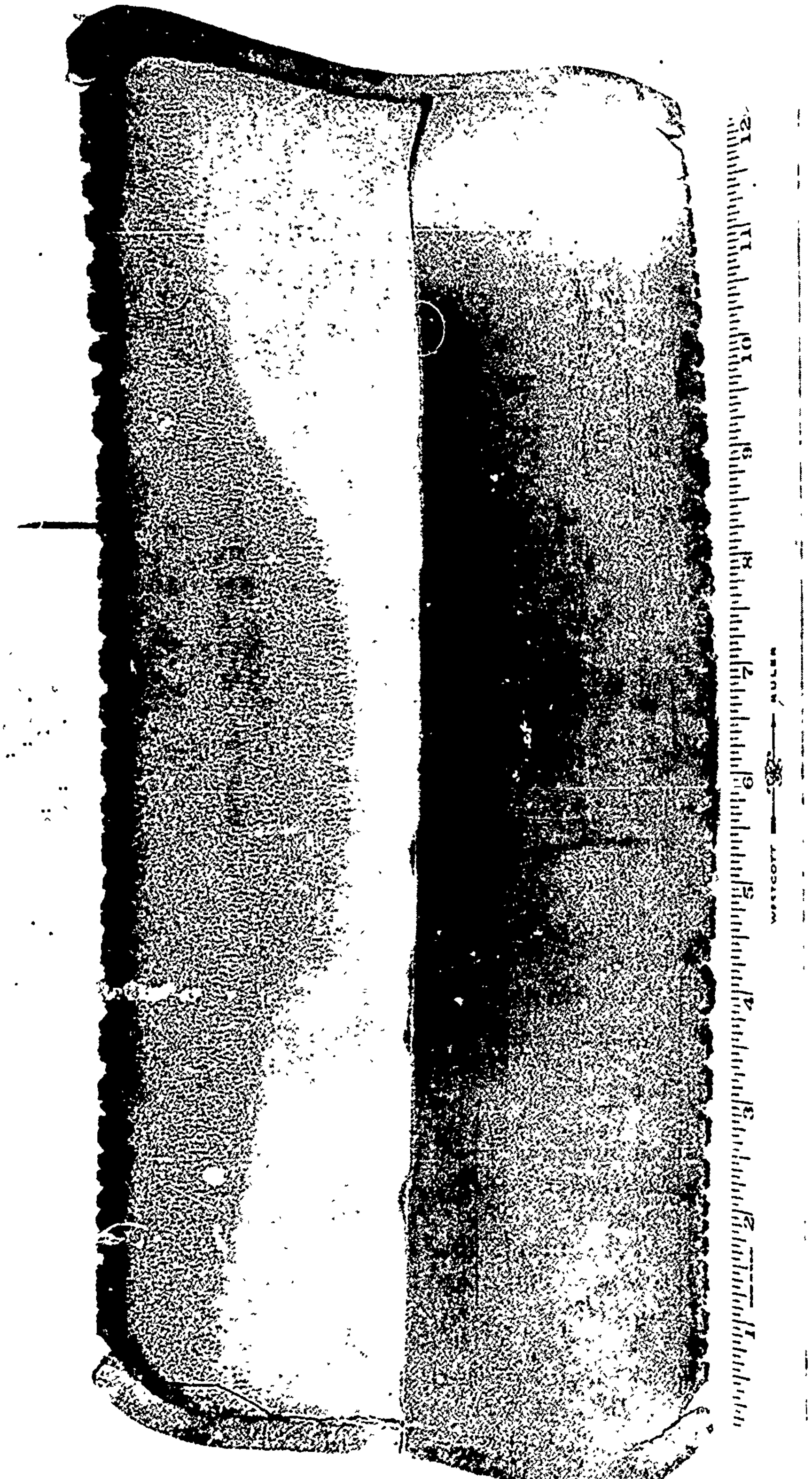


Figure 46 - Hot Rolled Sheet #35

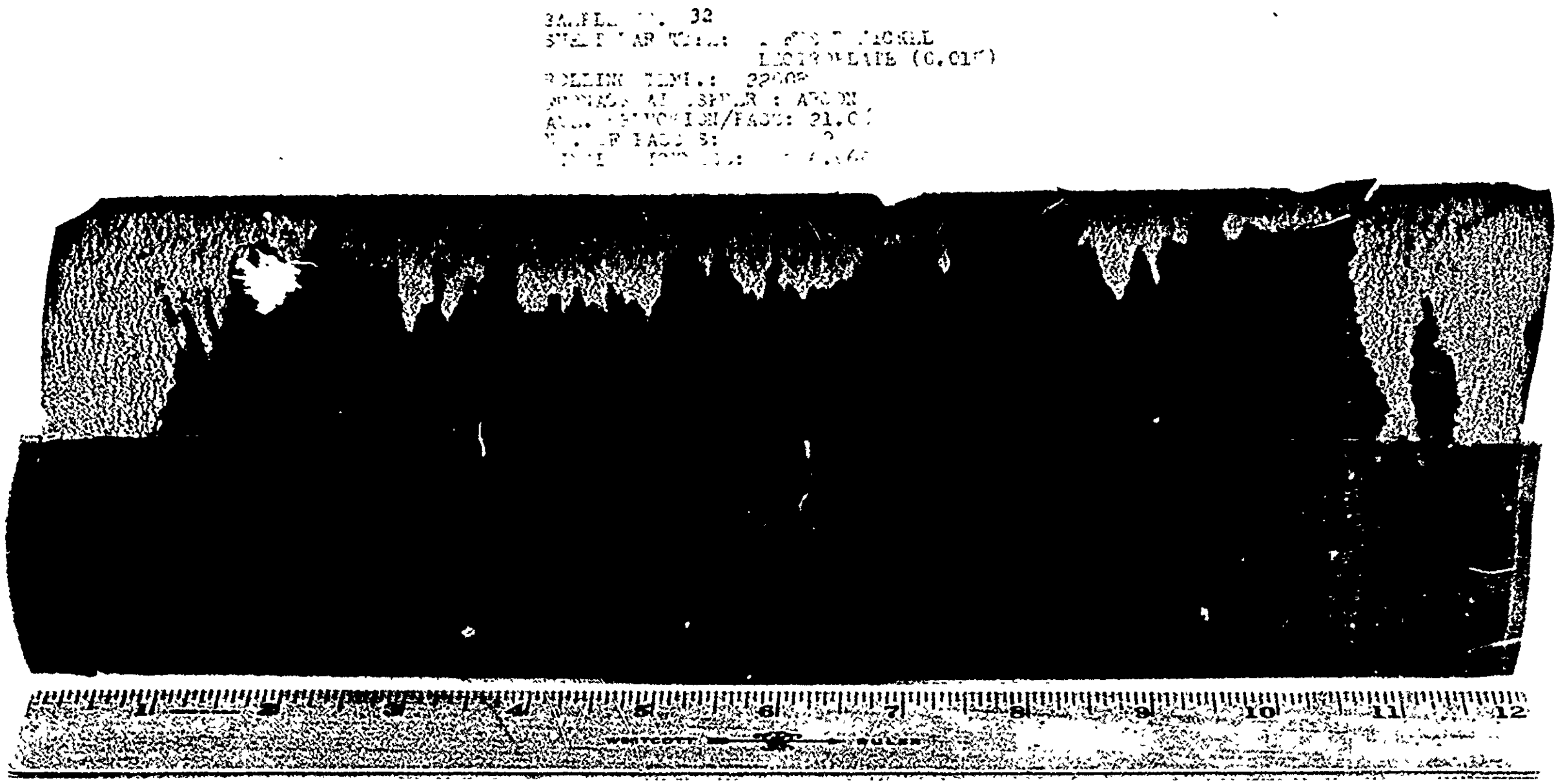


Figure 47 - Hot Rolled Sheet #32

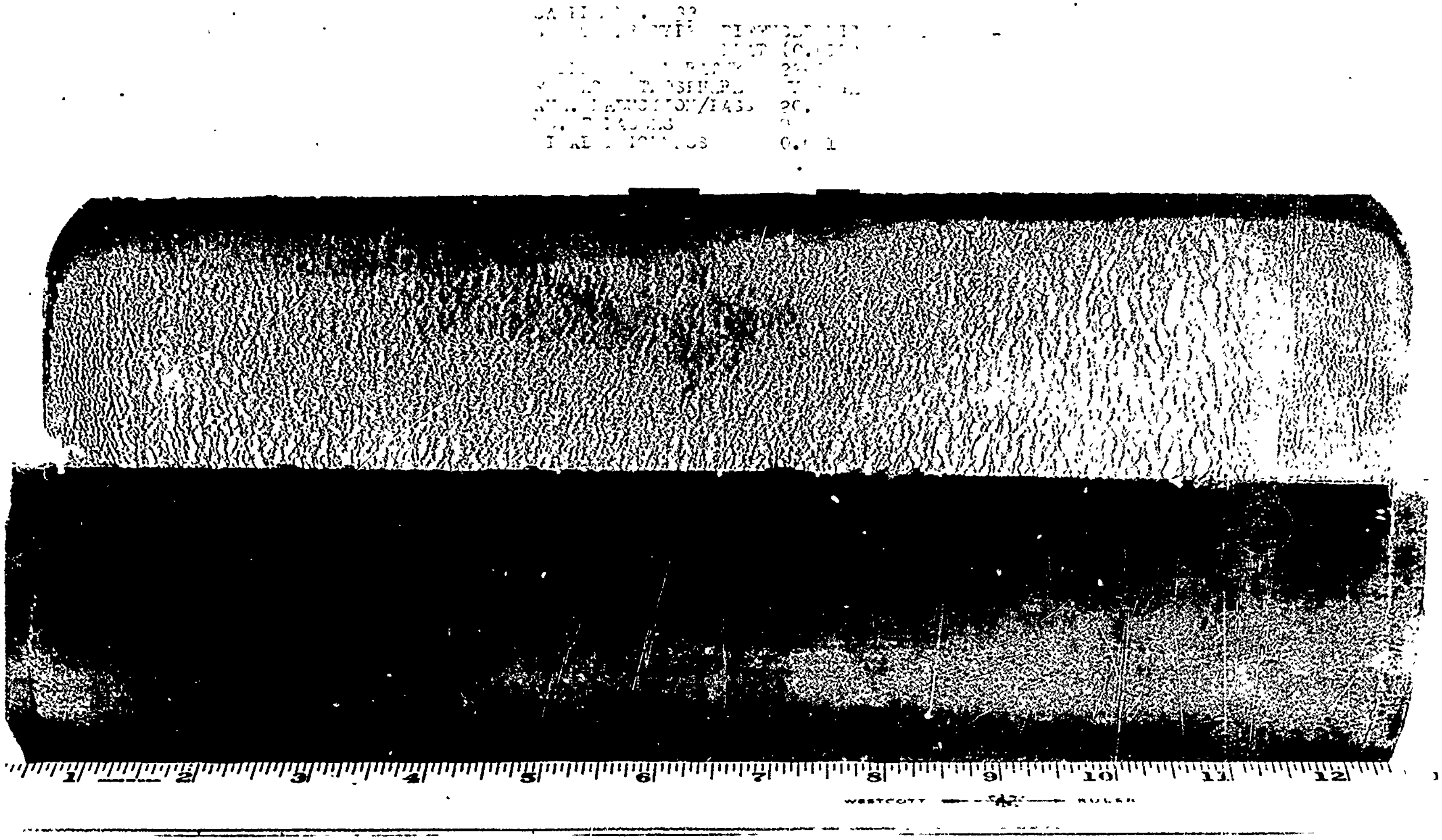


Figure 48 - Hot Rolled Sheet #33

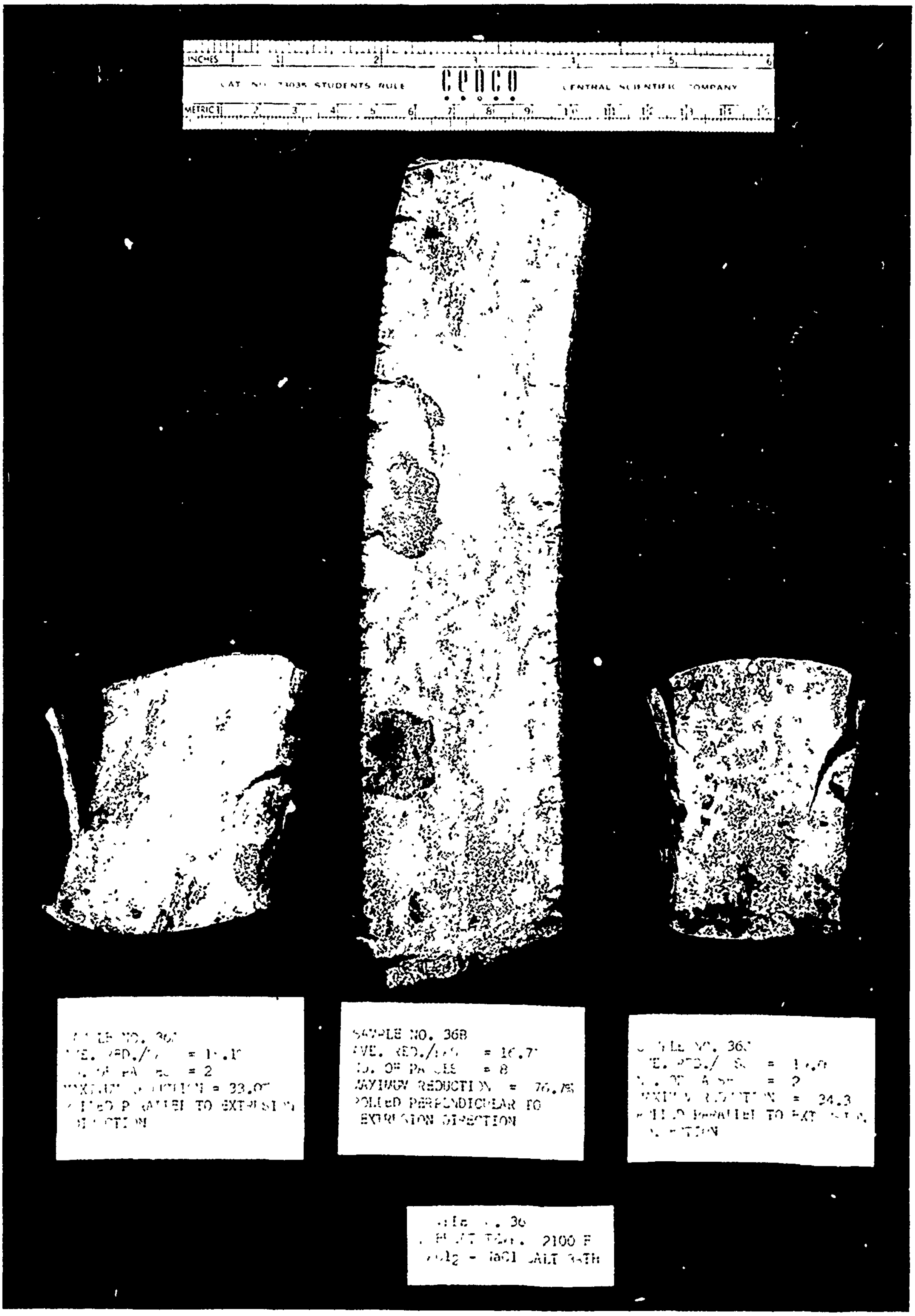


Figure 49 - Hot Rolled Sheet #36

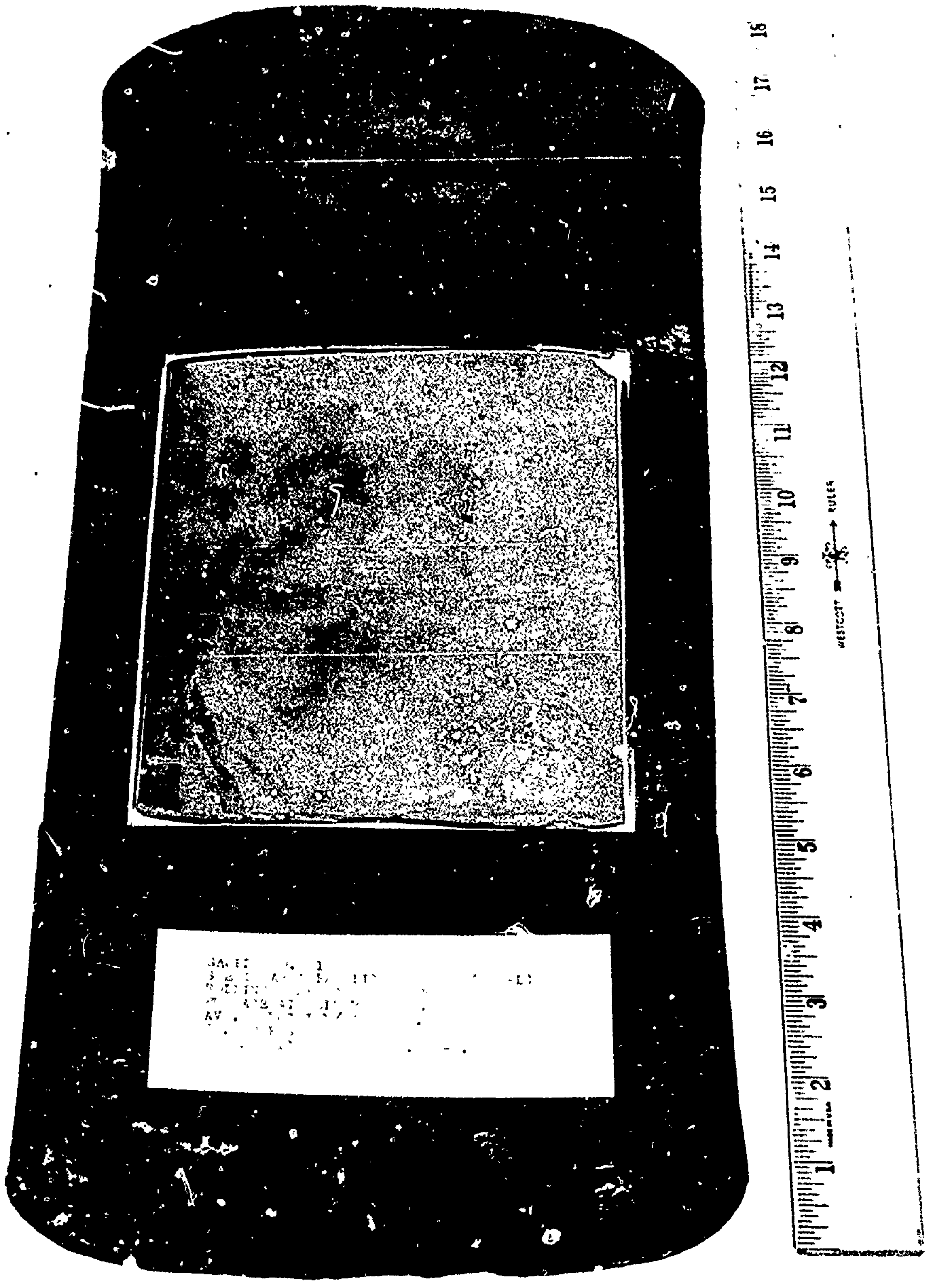


Figure 50 - Typical Hot Rolled Frame and Sheet After Cover Removal

PRINCIPAL ROLLING STUDIES

The major objective of the combined preliminary and principal rolling studies was to develop a sheet rolling procedure which would provide the lowest ductile-to-brittle tensile transition temperature in finished sheet. The discussion which follows summarizes the experimental rolling trials and metallurigical evaluation carried out to determine optimum rolling techniques.

Hot Rolling Trials

A series of 27 hot rolling experiments was performed to determine optimum working characteristics of extruded sheet bars. Framed sheet bars were rolled at temperatures ranging from 1800 to 2300° F, using roll pass reductions of 15 to 40 percent, to produce sheet samples having 70 to 85 percent total reduction. Rolling variables were evaluated on the basis of rollability, microstructure, hardness, surface condition and room temperature tensile properties. These rolling trials are summarized in Table 21 in the Appendix.

Rolling and Surface Characteristics

All sheet bars rolled to .060" experienced cover plate splits at the interface between sheet bar and frame which resulted in discoloration of sheet surfaces. Splitting generally did not occur until the last one or two passes, and the thin oxidized surface layers were easily removed by pickling. In nearly all cases, cover plates were observed to bubble during the rolling sequence indicating air entrapment. Some difficulty was experienced in removing cover plates from the rolled sheet although there was no apparent damage to sheet surfaces as a result of this slight bonding. All but three rolled framed assemblies were annealed for one half hour at the rolling temperature after the last roll pass as a precaution against cracking due to frame contraction.

Only two sheets, #38 and #41, both rolled at 1800° F, showed evidence of surface cracks or defects. These appeared to have cracked in the frame during cooling. Hardness measurements indicated that an annealing temperature of 1800° F between passes and after the final pass was not sufficient to relieve stresses induced during rolling. Several rolled frame assemblies were successfully flattened after the last pass under a forge hammer to provide flat material for the preparation of tensile bars. A typical rolled frame assembly is shown in Figure 49, and two pickled sheets are shown in Figure 51.

Average core nitrogen contents were found to be in close agreement with starting sheet bar values. This indicates that the frame enclosure technique offered adequate protection against contamination during rolling. The following nitrogen contents were determined, by Micro-Kjeldahl analysis, from hot rolled sheet specimens after various annealing treatments:

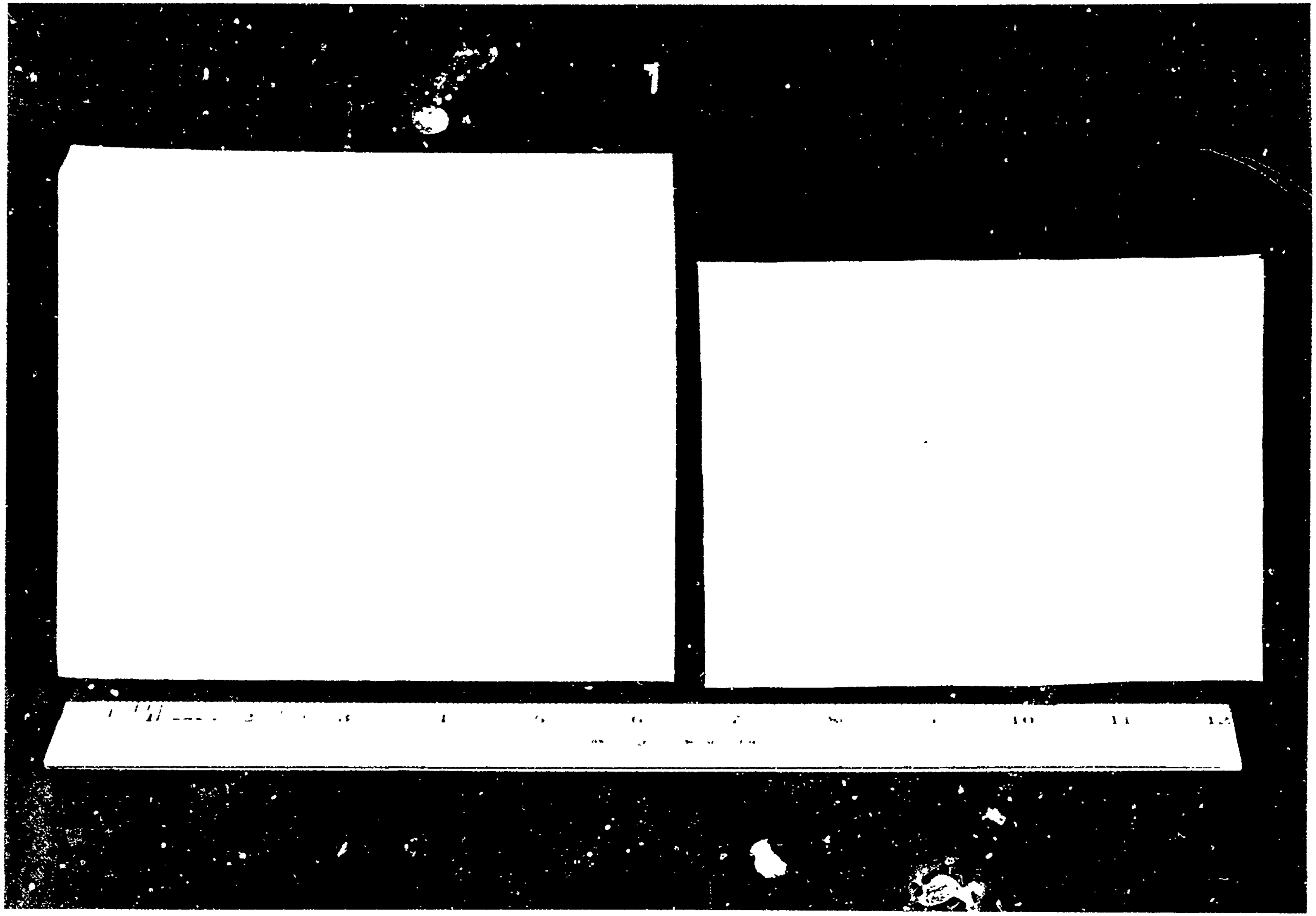


Figure 51 - Typical Sheets - Hot Rolled & Pickled

Sheet Sample	Sample History	Nitrogen Content, PPM
56	Annealed in Frame Only	60
57	Re-annealed @2200° F in Wet H_2	70
62	Re-annealed @2200° F in Vacuum	90
As Extruded (861)	Annealed @2200° F in Vacuum	80

Microstructure and Hardness Evaluation

The photomicrographs shown in Figures 52 and 53 provide a comparison of as-rolled and annealed microstructures for various hot rolling temperatures. The as-rolled structure resulting from the 1800° F rolling temperatures shows a considerable amount of retained work. An annealing temperature of 2000° F for one half hour was required to provide a uniform equiaxed grain structure. Rolling temperatures of 1900° F and 2200° F produced a coarse grain annealed structure possibly due to a secondary recrystallization phenomenon. A somewhat smaller grain size was obtained with a 2300° F rolling and annealing temperature. Initial rolling at 2200° F followed by a final 50 percent reduction at 1800° F provided a grain size similar to straight 1800° F rolling. Figures 54 and 55 provide a comparison of straight rolled and cross rolled microstructures for both 2000° F and 2200° F rolling temperatures. Cross rolled structures are nearly identical to the straight rolled in both longitudinal and transverse sections. The hardness data presented in Figure 56 and Table 7 compare closely with observed changes in microstructure. A one half hour anneal at the rolling temperature produced a noticeable softening even for sheet rolled at 2200° F. It can be seen from Table 7 that a one hour anneal at the rolling temperature provided an additional hardness reduction.

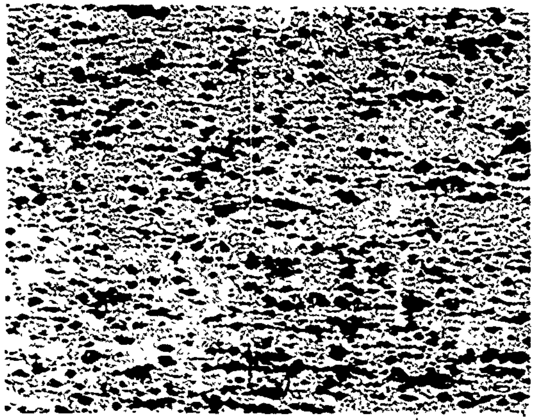
Room Temperature Tensile Properties

A majority of the hot rolled sheets were re-annealed for one half hour in a hydrogen atmosphere after frame removal and pickling in order to obtain minimum hardness prior to the evaluation of tensile properties. These sheets were inadvertently contaminated during the annealing cycle due to a malfunctioning gas dryer system. An adherent oxide coating was developed which could not be removed by pickling or vapor blasting. Tensile properties could not be evaluated therefore, without a costly grinding procedure to remove contaminated surface layers. A total of 28 flat tensile specimens were prepared however, by surface grinding and the tensile test results are tabulated in Table 8 along with data from sheets which were not contaminated. Test data from two flat tensile specimens taken from extruded material are included for comparison.

Several tensile specimens failed prematurely at the locating holes although care was taken to deburr and chamfer all sharp edges after drilling and grinding. As a consequence, later specimens were prepared according to the design shown previously in Figure 8. Initial tests were performed on tensile specimens which were not re-annealed after flattening and grinding. It was later learned that both surface grinding and flattening tended to work harden surface layers and impair tensile properties. Note the flat bar tensile data for extruded and annealed material (extrusion 845-3). Elongations of 9 percent and 14.6 percent in the ground and sanded condition are considerably lower than the normal 19-24 percent measured for polished round bars.

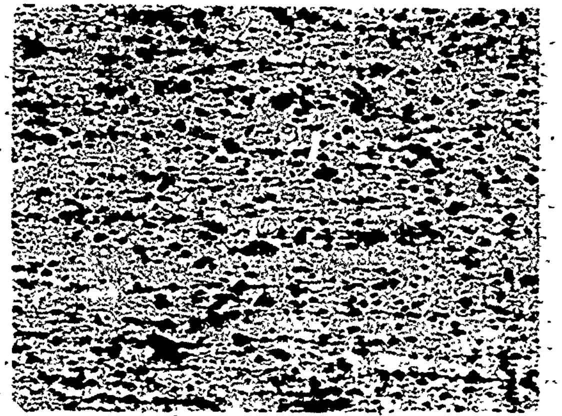
The best property data were obtained from pickled specimens which had not been flattened and/or ground. These specimens showed an appreciable room temperature

P-44



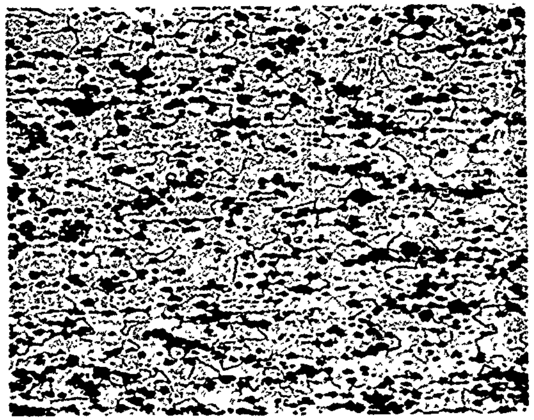
H.R. #38
Rolled @1800 F
As-Rolled

P-44



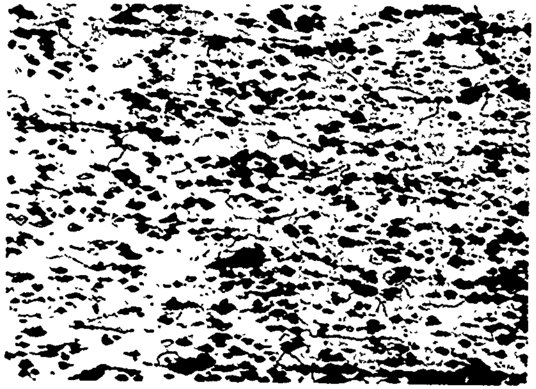
H.R. #41
Rolled @1800 F
Annealed 1800 F

P-46



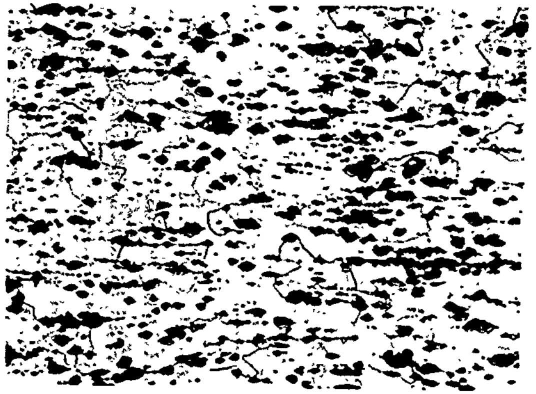
H.R. #38
Rolled @1800 F
Annealed 2000 F

P-94



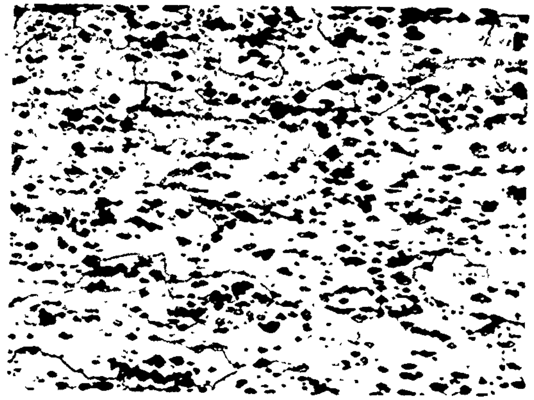
H.R. #81
Rolled @1900 F
As-Rolled

P-97



H.R. #77
Rolled @1900 F
Annealed 1900 F

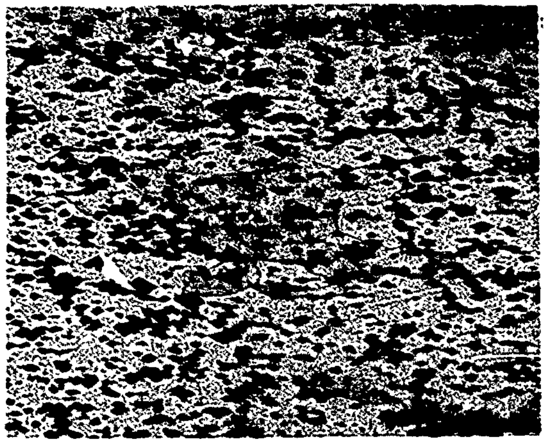
P-94



H.R. #79
Rolled @1900 F
Annealed 2200 F

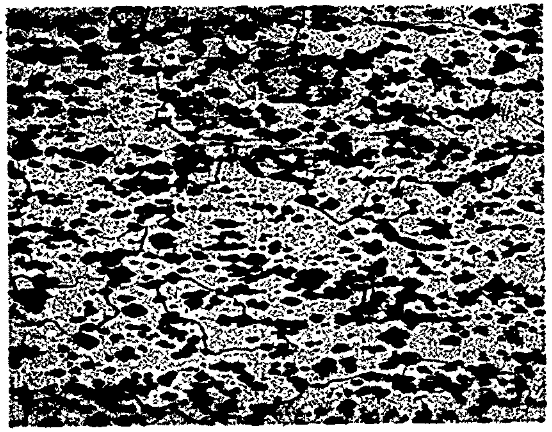
Figure 52 - Comparison of Hot Rolled and Annealed Microstructures (Magnification 125X)

O-997



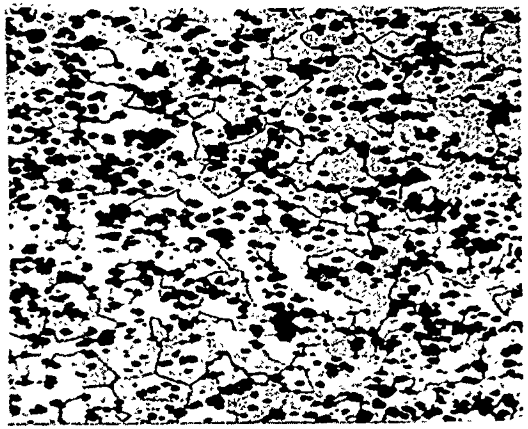
H.R. #31
Rolled @2200 F
As-Rolled

P-15



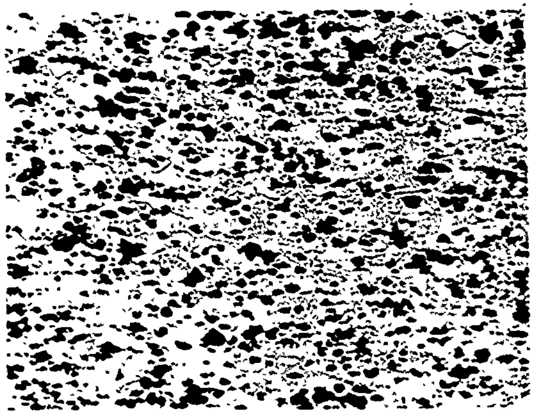
H.R. #31
Rolled @2200 F
Annealed 2200 F

P-136



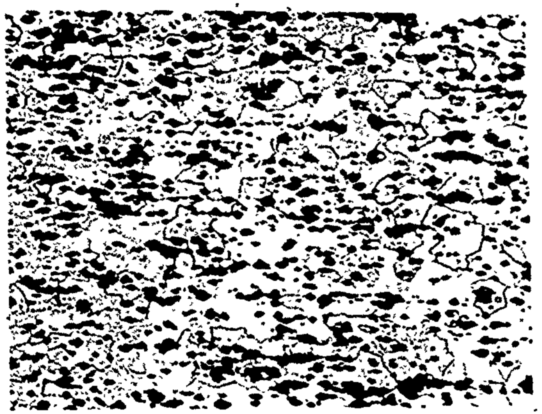
H.R. #49
Rolled @2300 F
Annealed 2300 F

P-93



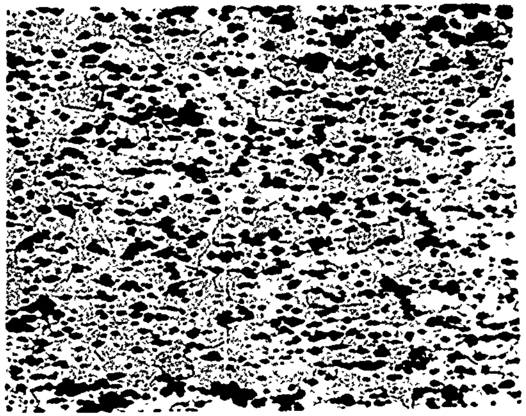
H.R. #70
Rolled @2200/1800 F
As-Rolled

P-97



H.R. #71
Rolled @2200/1800 F
Annealed 2000 F

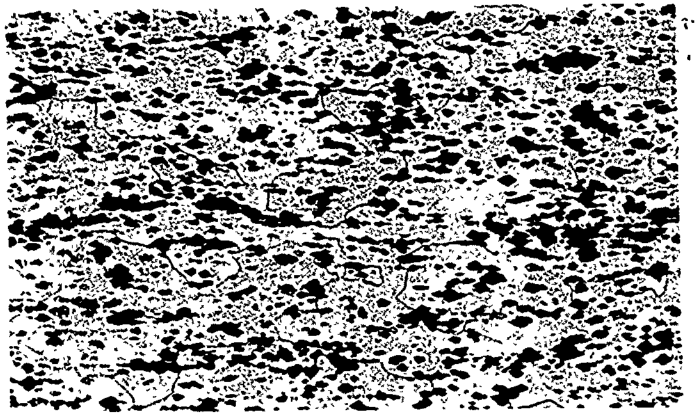
P-93



H.R. #73
Rolled @2200/1800 F
Annealed 2200 F

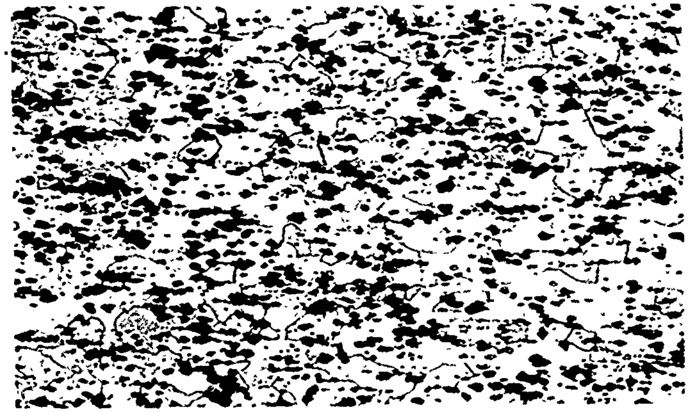
Figure 53 - Comparison of Hot Rolled and Annealed Microstructures (Magnification 125X)

P-27



H.R. #46 Longitudinal

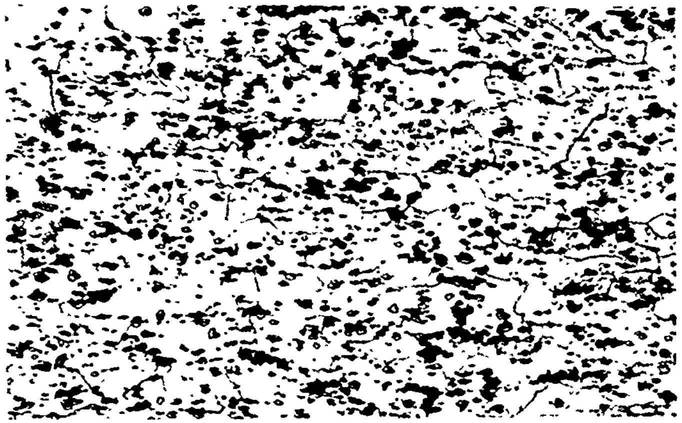
P-133



H.R. #51 Longitudinal

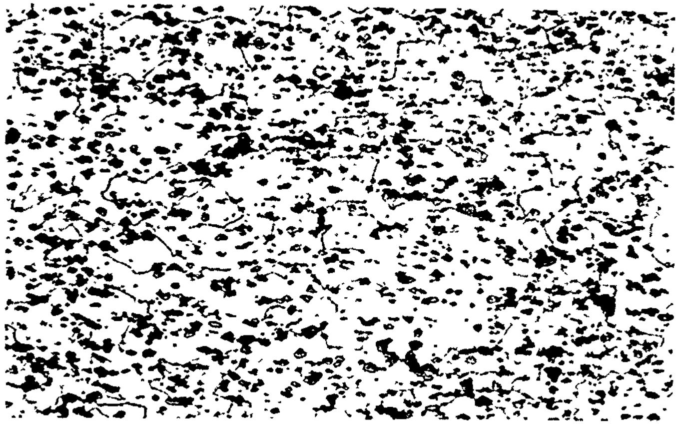
Straight Rolled (Magnification 125X) Cross Rolled 43%

P-27



H.R. #46 Transverse

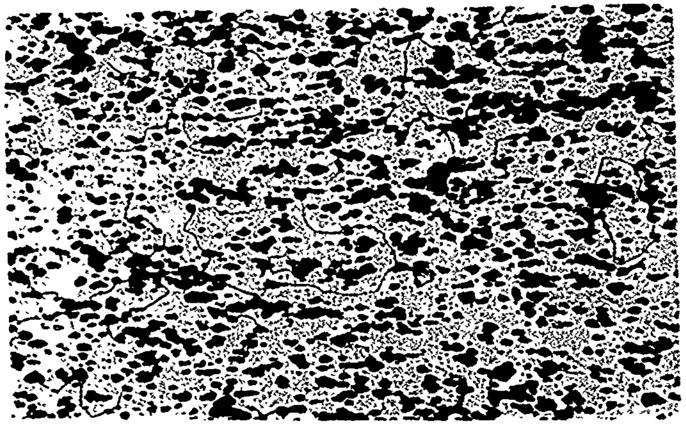
P-133



H.R. #51 Transverse

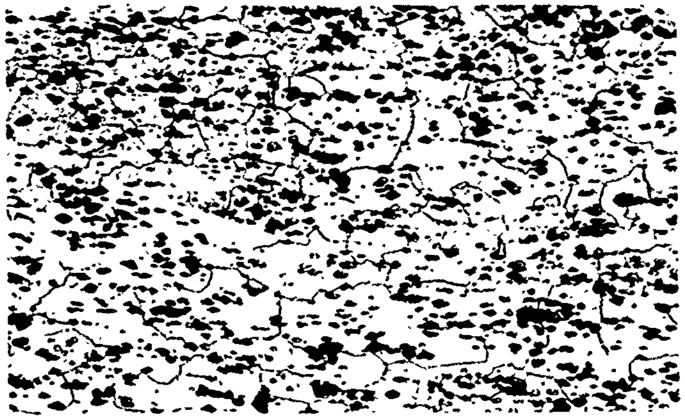
Figure 54 - Comparison of Straight and Cross Rolled Microstructures Rolled @2000 F - Annealed @2000

P-27



H.R. #47 Longitudinal

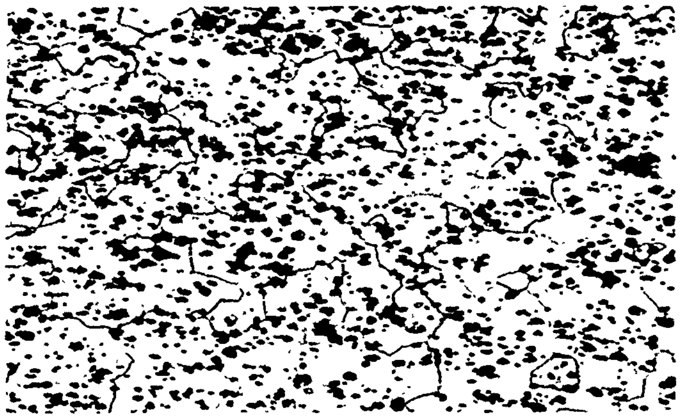
P-135



H.R. #52 Longitudinal

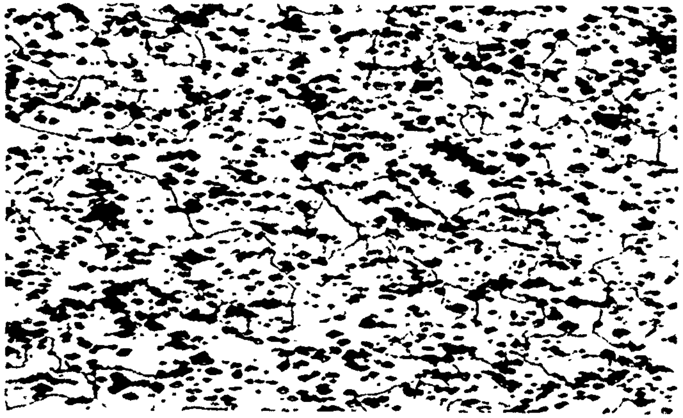
Straight Rolled (Magnification 125X) Cross Rolled 52%

P-27



H.R. #47 Transverse

P-135



H.R. #52 Transverse

Figure 55 - Comparison of Straight and Cross Rolled Microstructures Rolled @2200 F - Annealed @2200 F

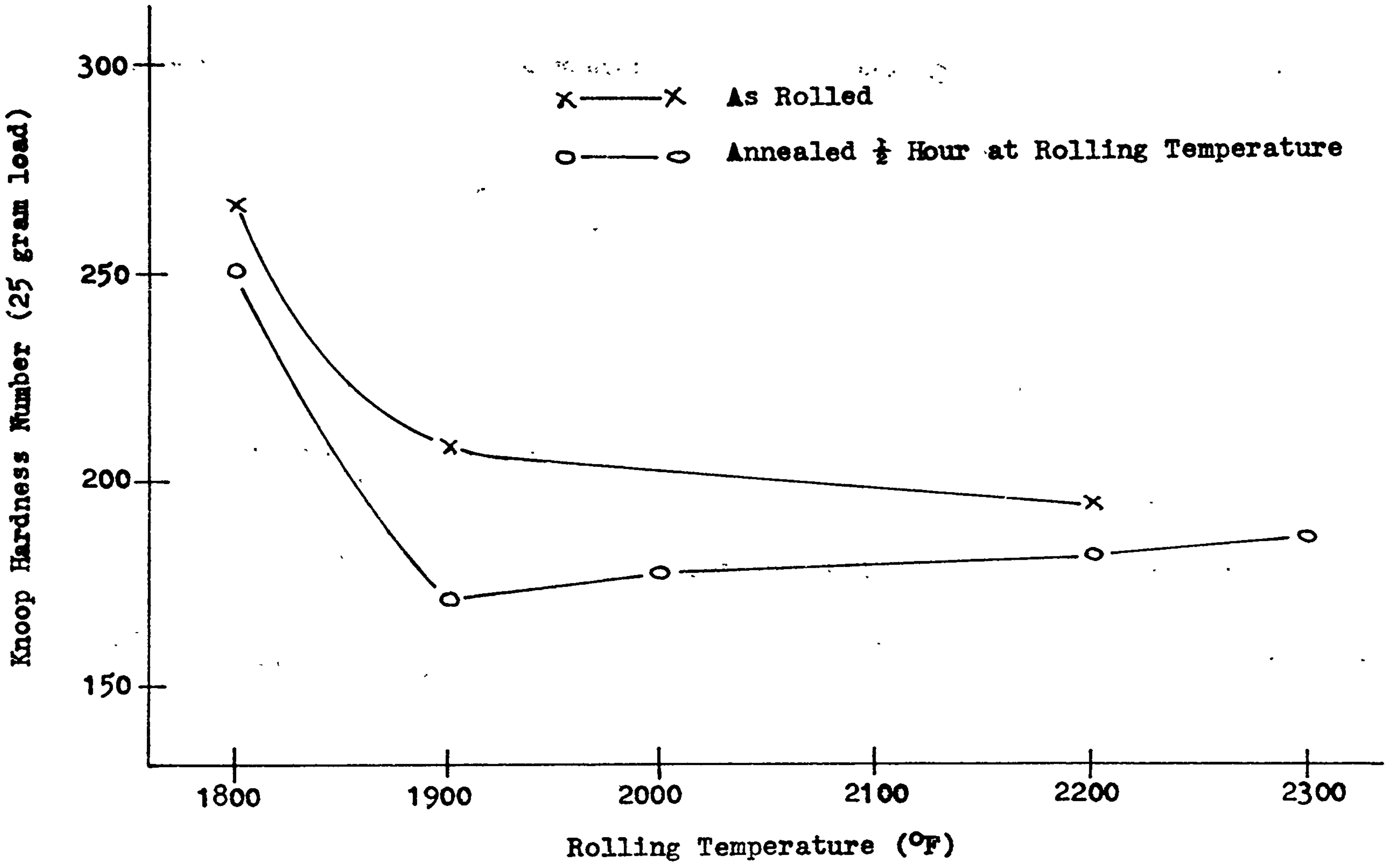


Figure 56 - Effect of Rolling and Annealing Temperature on the Hardness of Hot Rolled Sheet

Table 7. Effect of Rolling and Annealing Variables on Hardness of Hot Rolled Sheet

Rolling Trial Number	Rolling Temperature, F	Intended Reduction Per Pass, Percent	Total Reduction, Percent	Annealing Temperature, F	Hardness: ½ Hr. Anneal Knoop(a)	Hardness: ½ Hr. Anneal Rockwell 45 T(b)	Hardness: 1 Hr. Anneal Rockwell 45 T(b)
38	1800	15	82.0	As Rolled	271	70.0	-
41	1800	20	82.3	1800	250	67.5	58.5
38	1800	15	82.0	2000	217	57.0	56.0
81	1900	20	77.5	As Rolled	205	60.0	-
77	1900	20	78.5	1900	169	50.0	-
79	1900	20	78.0	2200	188	51.0	-
39	2000	15	83.3	2000	-	51.0	-
40	2000	20	82.5	2000	-	55.0	46.5
46	2000	25	82.2	2000	174-151(TR)	53.0	50.0
51	2000(c)	25	86.6	2000	195-190(TR)	53.0	-
54	2000	25	72.5	2000	-	43.5	-
31	2200	20	81.0	As Rolled	192	58.0	-
31	2200	20	81.0	2000	155	49.0	46.5
42	2200	20	82.6	2200	-	51.5	-
43	2200	15	84.0	2200	-	52.0	-
47	2200	25	82.0	2200	180-164(TR)	53.5	47.5
52	2200(d)	25	84.6	2200	172-193(TR)	53.0	-
44	2200	30	82.4	2200	-	53.5	47.0
55	2200	25	73.3	2200	-	50.0	-
56	2200	28	72.4	2200	-	49.0	43.5
57	2200	28	72.0	2200	-	48.0	-
49	2300	30	86.6	2300	182-183(TR)	52.0	-
50	2300	40	85.7	2300	-	51.0	-
70	2200/1800	30/20	78.0	As Rolled	280	67.5	-
71	2200/1800	30/20	78.0	2000	158	51.0	-
73	2200/1800	30/20	78.0	2200	200	51.0	-

(a) 25 gram load, average of three (3) readings. Longitudinal sections unless noted (TR), transverse to final roll direction.

(b) Average of ten (10) readings. Major load applied 10 seconds. Surfaces sanded 4/0 paper.

(c) Cross rolled last 43%.

(d) Cross rolled last 52.6%.

Table 8. Effect of Rolling and Annealing Procedure on the Room Temperature Tensile Properties of Hot Rolled Sheet

Sheet Specimen Number	Rolling Temperature, F	Annealing Temperature, F	Annealing[a] Time Hour	Atmosphere	Ultimate Strength, 1000 PSI	Yield Strength,[b] 1000 PSI	Elongation in 1 Inch, Percent[c]
41-1	1800	2200	½	Wet H_2	43.1	29.6-27.2	3.5
41-2	1800	2200	½	" "	41.6	31.5-29.4	2.6
46-1	2000	2000	½	Wet H_2	Failed at Hole		5.2
46-2	2000	2000	½	" "	44.5	26.5	
31-2	2200	2000	½	Vacuum	Failed at Hole		12.5
31-3	2200	2000	½	Dry H_2	44.5	19.3	9.4
44-1	2200	2200	½	Wet H_2	48.3	29.3	6.0
44-2	2200	2200	½	" "	Failed at Hole		
47-1	2200	2200	½	" "	45.2	31.1	2.9
47-2	2200	2200	½	" "	Failed at Hole		
56-1	2200	2200	None	Frame	43.2	24.4-22.4	7.9
56-2	2200	2200	"	"	35.6	27.6-22.5	2.8
56-3	2200	2200	"	"	Failed at Hole		
56-5	2200	2200	½	Wet H_2	36.5	25.8	2.5
57-1	2200	2200	½	Vacuum	38.5	24.5	3.7
57-2	2200	2200	½	"	39.6	24.4	4.0
57-4	2200	2200	½	Wet H_2	41.0	25.7	4.4
57-5	2200	2200	½	" "	41.1	25.8	4.1
57-6	2200	2200	½	" "	42.5	25.4	6.0
77-1	1900	1900	None	Frame	44.9	26.9	6.6[d]
77-2	1900	1900	"	"	46.1	25.9	9.2[e]
77-3	1900	1900	"	"	46.3	25.4	13.0[e]
77-4	1900	1900	"	"	45.0	25.1	9.8[e]
79-1	1900	2200	"	"	45.6	26.1	14.5[e]
79-2	1900	2200	"	"	47.0	27.3	14.7[e]
79-3	1900	2200	"	"	42.4	27.4	3.7[e]
75-1	2000	2000	"	"	45.6	28.2	5.0[d]
75-3	2000	2000	"	"	40.8	25.8	3.7[e]
75-4	2000	2000	"	"	44.3	25.5	7.5[e]
71-1	2200/1800	2000	"	"	49.2	28.2	6.6[d]
71-2	2200/1800	2000	"	"	47.6	26.2	15.0[e]
71-4	2200/1800	2000	"	"	46.2	25.1-24.0	15.0[e]
845-3-1	Extruded	2000	2	Vacuum	45.2	27.6-26.6	14.6
845-3-2	Extruded	2000	2	"	45.6	26.0-25.9	9.0

(a) All sheets except #31 previously annealed for ½ hour in the frame.
(b) Upper and lower yield reported where observed. Others 0.2% offset.
(c) Surfaces ground and sanded with 4/0 grit paper unless otherwise noted.
(d) Surfaces as rolled.
(e) Surfaces as pickled.

tensile elongation even though the grain size was abnormally large. Although it was not possible to correlate rolling procedure with tensile properties, the tensile work accomplished during this phase of the program provided considerable background information on the specimen configuration and preparation required for a consistent and reproducible test results.

Hot-Warm Rolling Trials

Eleven sheets were rolled to further evaluate hot rolling procedures and annealing treatments, and to obtain preliminary information on the warm rolling of hot rolled sheet. These rolling trials are summarized in Table 22 in the Appendix. Hot rolled sheets were examined visually for surface defects and conditioned for warm rolling. The resulting warm rolled sheets were evaluated on the basis of rollability and surface characteristics, microstructure, hardness, and room temperature tensile properties.

Rolling and Surface Characteristics

Sheets #58 and #59, when annealed in wet hydrogen, developed an adherent oxide coating which could not be removed by pickling. These sheets were successfully warm rolled to 49 percent and 46 percent reduction respectively with only slight edge cracking. Extensive cracking occurred however, when a "no-reduction" flattening pass was attemped. V-shaped markings were visible on sheets #72 and #74 after hot rolling indicating that there may have been surface cracks in the machined sheet bars. These markings, although faintly visible after warm rolling, did not impair rollability of the sheets. A "finger-print" type lamination, visible on sheet #72 after hot rolling, was still visible after warm rolling. All the remaining hot rolled sheets were free of surface defects. The edges of sheet #66 were left in the rough pickled condition after warm rolling and edge splits developed during the first roll pass. The split ends were removed with a band saw and edge splits again developed with continued rolling. Rolling without edge cracks was finally accomplished when sawed edges were sanded, in the direction of rolling, with a 1/0 grit abrasive paper on a belt sander. All other sheets were prepared with sanded edges and were warm rolled with only slight edge cracking.

Microstructure and Hardness Evaluation

As rolled and annealed microstructures are shown in the photomicrographs of Figures 57, 58 and 59. An annealing temperature of 2000° F was adequate to provide completely recrystallized structures in all cases. Variations in hot rolling procedure had no significant effect on the microstructure or hardness of warm rolled sheet. Microstructures of straight rolled and cross rolled sheets are compared in Figures 60 through 63. This series of photomicrographs traces the recrystallization process with increasing annealing temperature. The early stages of recrystallization were apparent after annealing at 1600° F. Recrystallization was nearly complete after annealing at 1800° F, and fully recrystallized structures were obtained after annealing at 2000° F. The hardness data shown in Table 9 compare closely with these changes in microstructure. Straight rolled and cross rolled structures were identical after annealing.

Room Temperature Tensile Properties

Data from 28 tensile tests are reported in Table 10. Warm rolled sheets with 38 percent to 40 percent warm work showed appreciable room temperature tensile ductility

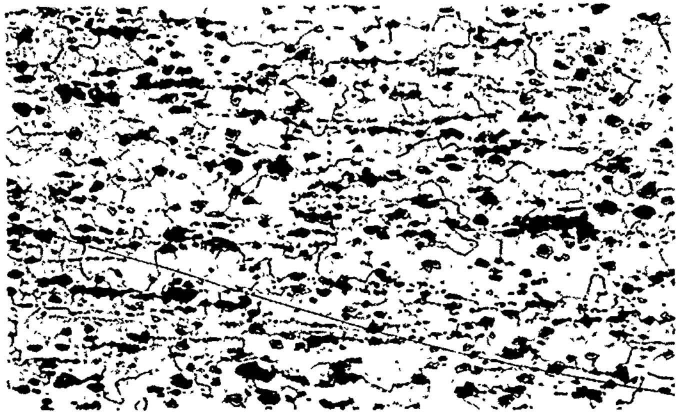
P-155

H.R. #78 W.R. #85

Hot Rolled
1900 F

Annealed
1900 F

P-155



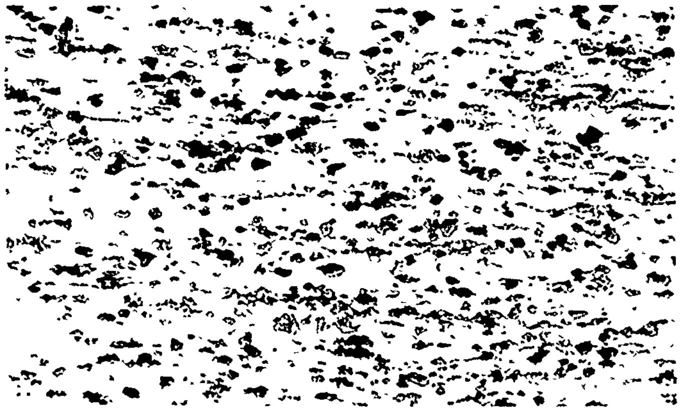
H.R. #78 W.R. #85

After Warm Rolling

(Magnification 125X)

Warm Rolled and
Annealed @2000 F

P-155



H.R. #80 W.R. #86

Hot Rolled
1900 F

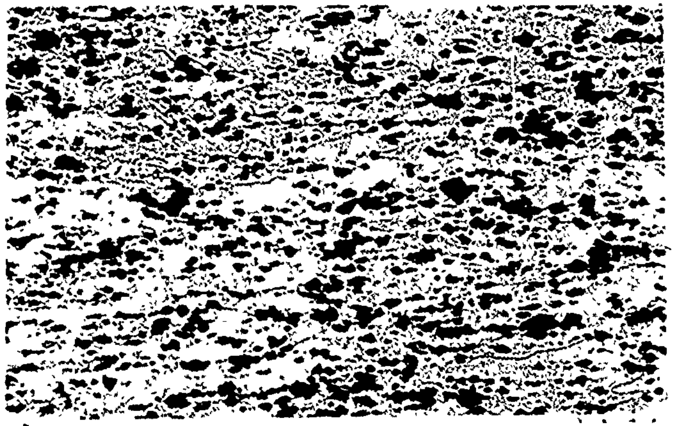
Annealed
2200 F

P-155

H.R. #80 W.R. #86

Figure 57 - Effect of Prior Hot Rolling Procedure on Warm Rolled Microstructure Warm Rolled 40 Percent @900 F

P-155

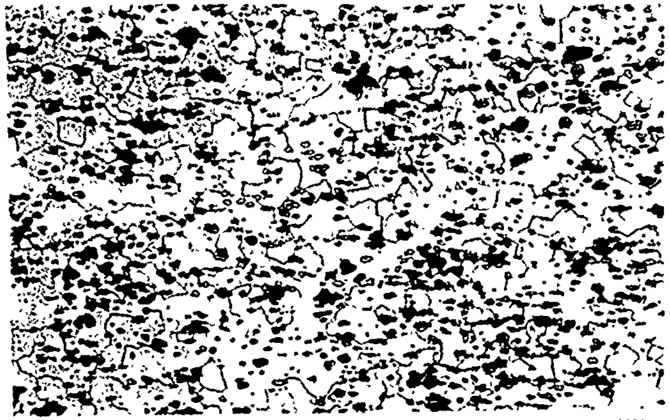


H.R. #76 W.R. #84

Hot Rolled
2200 F

Annealed
2000 F

P-156



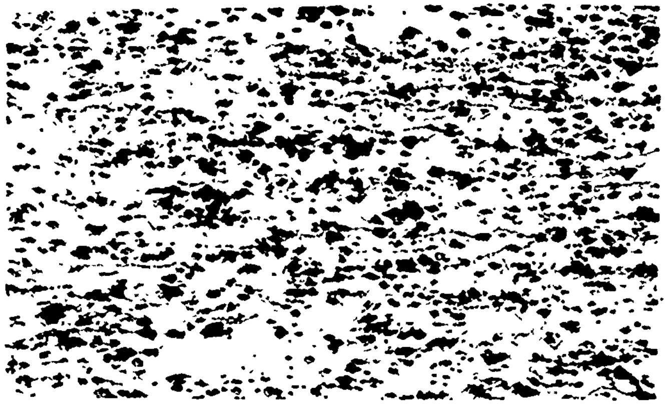
H.R. #76 W.R. #84

After Warm Rolling

(Magnification 125X)

Warm Rolled and
Annealed @2000 F

P-77



H.R. #60 W.R. #66

Hot Rolled
2200 F

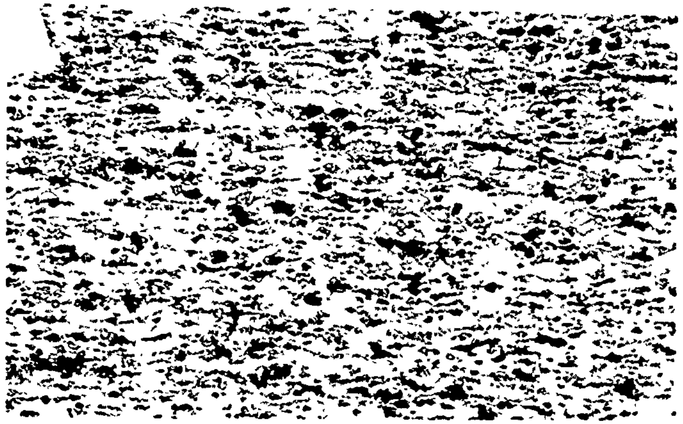
Annealed
2200/2000 F

P-77

H.R. #60 W.R. #66

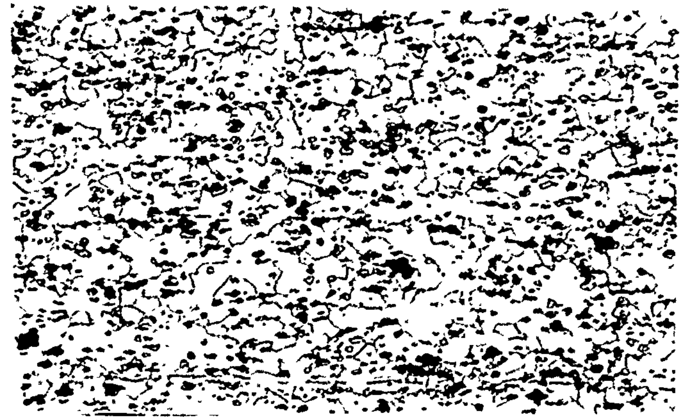
Figure 58 - Effect of Prior Hot Rolling Procedure on Warm Rolled Microstructure Warm Rolled 40 Percent @900 F

P-156



H.R. #72 W.R. #82A

P-156



H.R. #72 W.R. #82A

Hot Rolled 2200/1800 F

Annealed 2000 F

After Warm Rolling

(Magnification 125X)

Warm Rolled and Annealed @2000 F

P-156

H.R. #74 W.R. #83

Hot Rolled 2200/1800 F

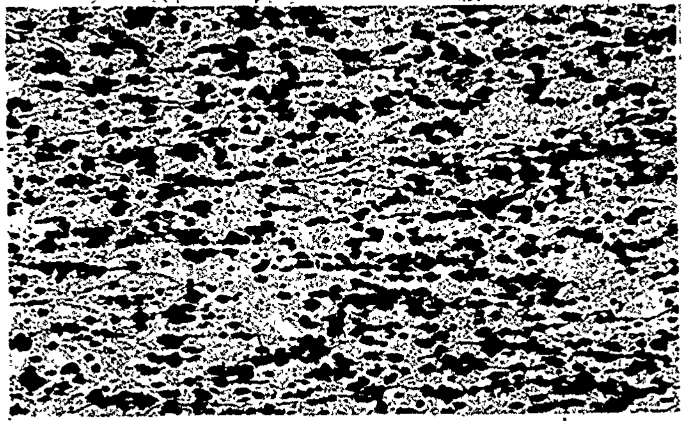
Annealed 2200 F

P-97

H.R. #74 W.R. #83

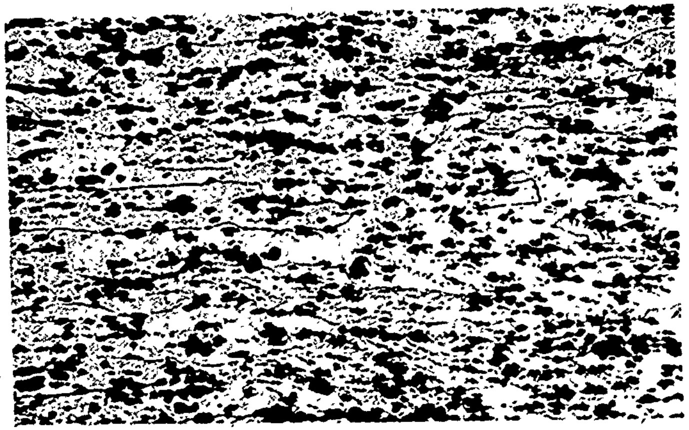
Figure 59 - Effect of Prior Hot Rolling Procedure on Warm Rolled Microstructure Warm Rolled 40 Percent @900 F

P-77



Longitudinal

P-78



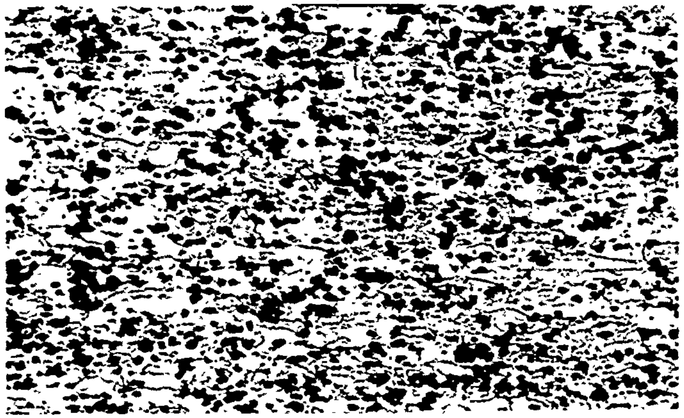
Longitudinal

Straight Rolled
W.R. #66

(Magnification 125X)

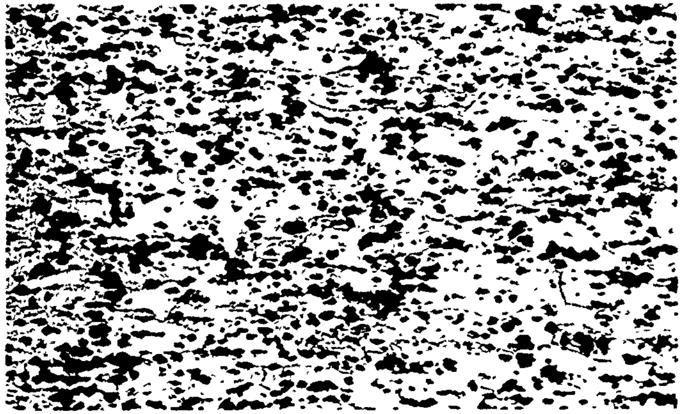
Cross Rolled
W.R. #69

P-77



Transverse

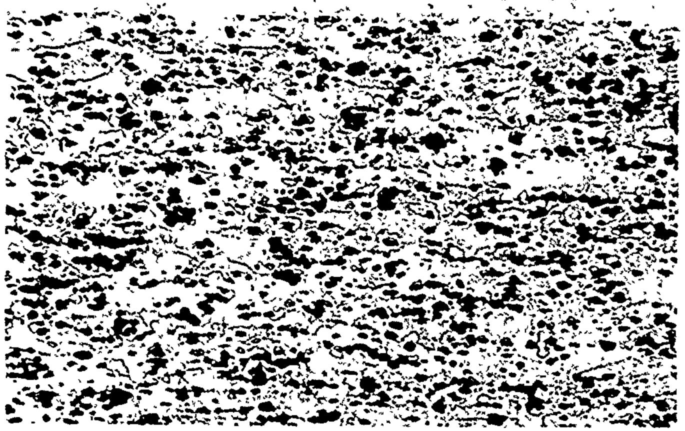
P-78



Transverse

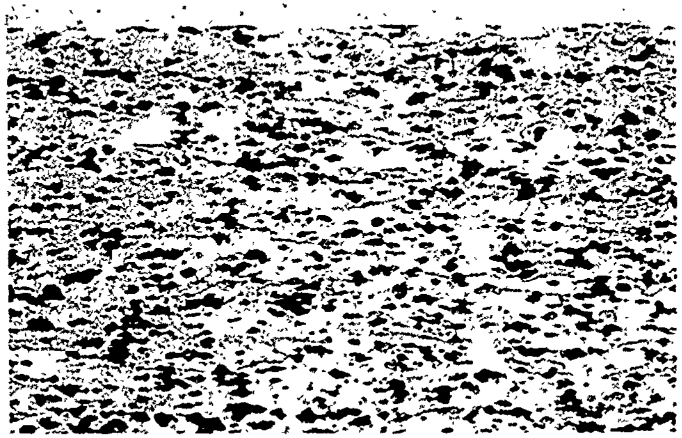
Figure 60 - As Rolled Microstructures of Cross Rolled and Straight Rolled Sheet Warm Rolled 900 F - 40 Percent Reduction

P-77



Longitudinal

P-78



Longitudinal

Straight Rolled
W.R. #66

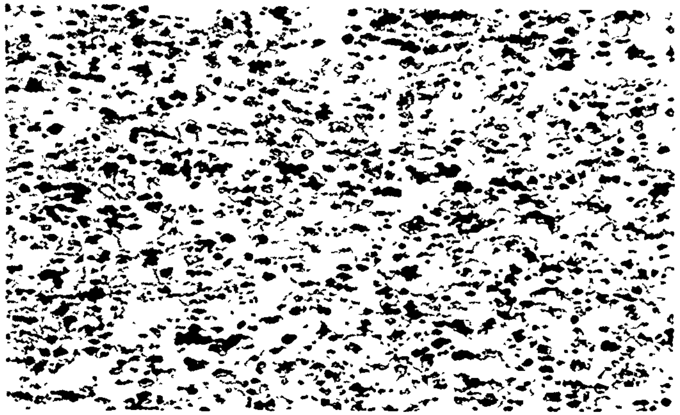
(Magnification 125X)

Cross Rolled
W.R. #69

P-77

Transverse

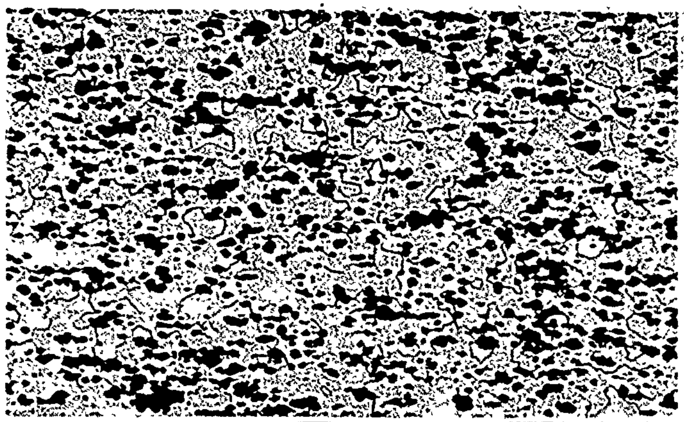
P-78



Transverse

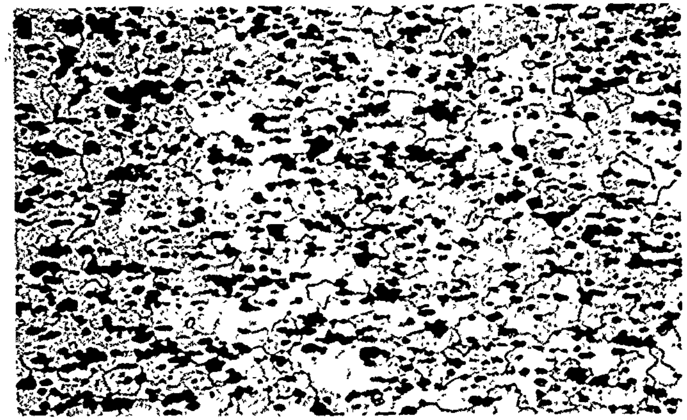
Figure 61 - Microstructures of Straight and Cross Rolled Sheet Annealed @1600 F - Warm Rolled 900 F - 40 Percent Reduction

P-77



Longitudinal

P-78



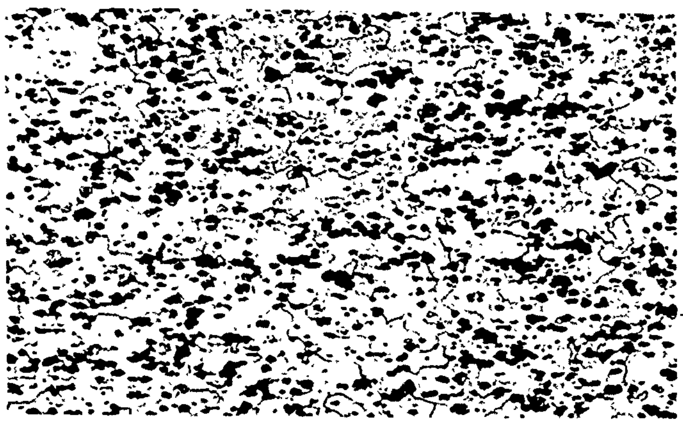
Longitudinal

Straight Rolled
W.R. #66

(Magnification 125X)

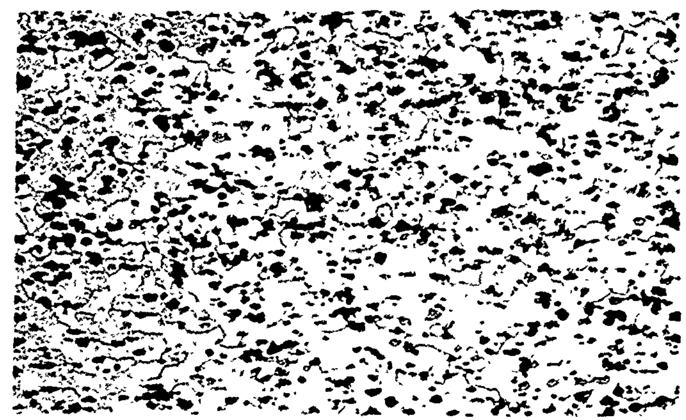
Cross Rolled
W.R. #69

P-77



Transverse

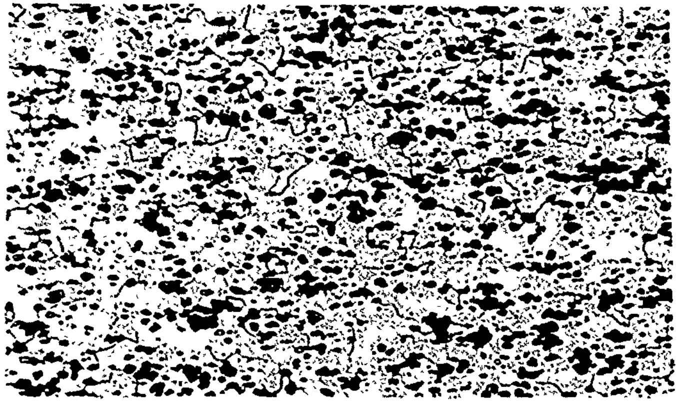
P-78



Transverse

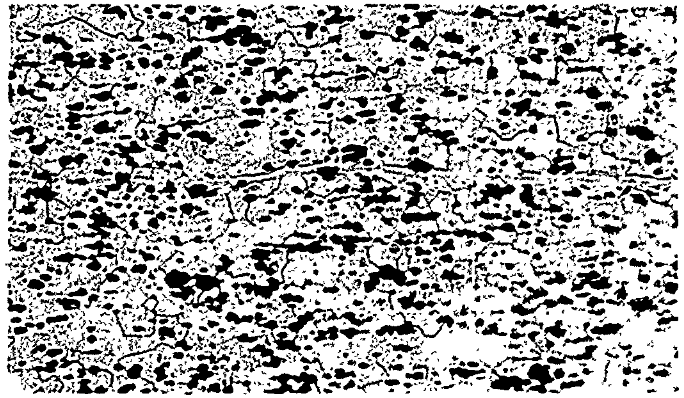
Figure 62 - Microstructures of Straight and Cross Rolled Sheet Annealed @1800 F - Warm Rolled 900 F - 40 Percent Reduction

P-77



Longitudinal

P-78



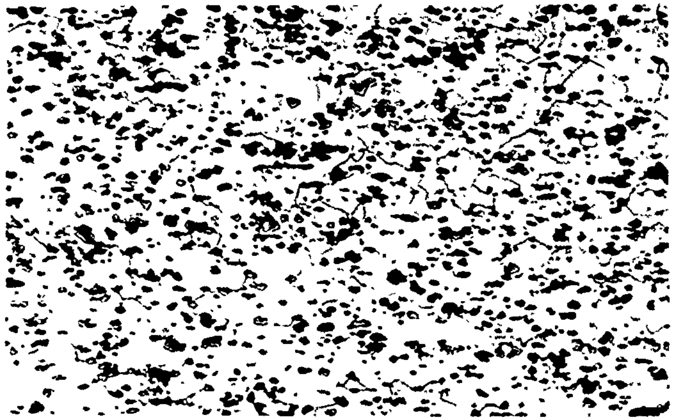
Longitudinal

Straight Rolled
W.R. #66

(Magnification 125X)

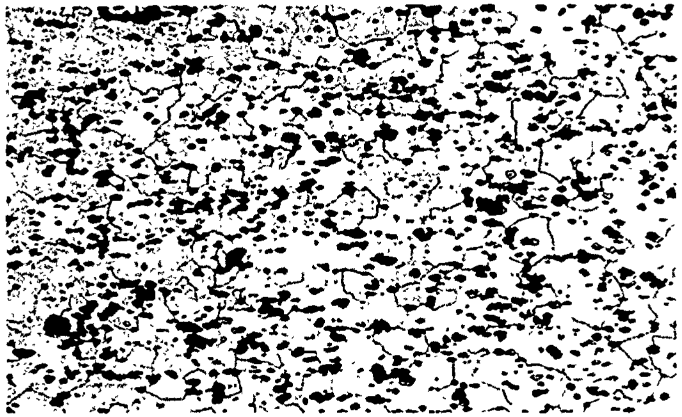
Cross Rolled
W.R. #69

P-77



Transverse

P-78



Transverse

Figure 63 - Microstructures of Straight and Cross Rolled Sheet Annealed @2000 F - Warm Rolled 900 F - 40 Percent Reduction

Table 9. Effect of Prior Hot Rolling and Annealing Procedure on the Hardness of Warm Rolled Sheet[a]

Warm Rolling Trial Number	Hot Rolling Temperature, F	Annealing Temperature After Hot Rolling, F	Annealing Temperature After Warm Rolling,[b] F	Hardness: Knoop 25 Gram Load	Hardness: Rockwell 45 T[c]
85	1900	1900	As Rolled	275	66.0
			2000	180	52.0
86	1900	2200	As Rolled	286	-
			2000	167	52.2
84	2200	2000	As Rolled	295	-
			2000	165	52.5
82A	2200/1800	2000	As Rolled	260	-
			2000	180	52.9
83	2200/1800	2200	As Rolled	273	66.0
			2000	183	-
66	2200	2200/2000	As Rolled	300	-
			1600	227	-
			1800	195	-
			2000	188	-
68	2200	2200/2000	2000	-	51.0

(a) All sheets finish rolled approximately 40 percent at 800°F.

(b) Vacuum annealed for 1/2 hour at the indicated temperature.

(c) Major load applied for 5 seconds.

Table 10. Effect of Prior Hot Rolling and Annealing Procedure on the Room Temperature Tensile Properties of Warm Rolled Sheet(a)

Warm Rolling Trial	Final Annealing Temp. °F	Final Annealing Time Hr.	Surface Condition(b)	Ultimate Strength 1000 PSI	Yield Strength(b) 1000 PSI	Elongation in 1 Inch, Percent	Number of Tests
				Average Longitudinal Tensile Properties After Indicated Anneal and Surface Treatment			
85	1450	2	AR	55.8	55.8	0	2
			EP	47.6	47.5	0	2
85	2000	½	AR	48.2	27.4	5.0	2
			EP	50.3	27.1-25.6	16.7	2
86	2000	½	AR	50.5	30.5-28.6	7.4	2
			EP	51.9	31.8-28.0	16.7	2
84	2000	½	AR	51.4	31.0-27.8	10.7	3
82A	2000	½	AR	54.8	31.6-30.9	9.8	1
			EP	54.3	31.4-29.6	16.0	2
67	2000	1(b)	AR	48.6	27.0-25.9	8.6	3
68	2000	½	AR	47.5	26.1	6.7	2
			P	48.3	26.0	10.0	2
69	2000	1(b)	AR	50.0	28.0-26.8	7.6	3

(a) All sheets warm rolled approximately 40% at 800°F.

(b) AR - As Rolled; EP - Electropolished; P - Pickled in Hydrochloric Acid.

(c) Upper and lower yield reported where observed. Others 0.2% offset.

(d) Tensile strips flattened in air at 1200°F and re-annealed prior to contour grinding.

in the fully recrystallized condition. Brittle behavior was observed for material warm worked 40 percent and stress relieved at 1450° F for two hours. The dependence of tensile elongation on surface condition is apparent. Electropolished samples provided consistently higher values than those obtained for as-rolled surfaces. It is significant, however, that 5 percent to 10 percent elongation was obtained from specimens with surfaces in the as-rolled condition. The tensile properties of warm rolled sheet did not appear to be affected by the prior hot rolling and annealing procedure. The data are inadequate, however, for a significant comparison. In general, it appears that surface condition and final annealing treatment, more than rolling procedure, will control the tensile properties of chromium composite sheet.

Warm Rolling Trials

The information gathered from the preceeding rolling investigations was used to select an optimum breakdown rolling procedure and to provide guide lines for the evaluation of warm rolling variables. The selected breakdown techniques consisted of hot rolling at 2200° F in a steel frame, with and without intermediate rolling at 1800° F, using roll pass reductions of 25 - 30 percent (20 percent for 1800° F rolling). Hot rolled sheets were annealed for 1/2 hour at 2200° F in the frame after the last roll pass and subsequently vacuum annealed for 1/2 hour at 2000° F after frame removal and pickling.

Flattening and shearing of the framed sheet, which was successfully carried out in earlier investigations, was found to be undesirable during the first ten warm rolling trials. The steel frame covers tended to adhere to sheet surfaces after the flattening operation causing the surfaces to become damaged on separation. Several sheets were badly cracked when an attempt was made to hot shear the frame edges. Both of these operations were discontinued and the rolling trials repeated with new sheet bars.

Additional difficulty was encountered during the hot rolling because of grinding cracks which were undetected in sheet bars prior to framing. All of the hot rolled sheets contained V-shaped markings in various degrees. Ten of the most severely marked sheets were discarded after hot rolling and replaced with rolled assemblies containing re-machined sheet bars. The less severely marked sheets were warm rolled successfully without further crack propagation. Defective surface areas were marked and discarded prior to obtaining tensile bars.

Conditioning of hot rolled sheets prior to warm rolling was carried out according to previously described procedures. Although frame covers were lightly bonded to sheet surfaces in some cases, separation was made without damaging the sheets. Release of the sheets from the frame was improved in later rolling trials by dusting each sheet bar with Thoria (ThO_2) prior to frame assembly.

Rollability

The warm rolling trials were made at 600, 900, and 1200° F using roll pass reductions ranging from 7 - 10 percent. Table 23 in the Appendix gives the details of each of the 27 successful trials. It can be seen that total hot rolling reductions were adjusted to provide for a finished sheet thickness of 0.050 inches after total warm rolling reductions of 8, 20, and 40 percent.

Sheets rolled at 600° F were less discolored than those rolled at the higher temperatures, and in several instances less roll deflection was obtained during 600° F rolling. These differences were slight, however, and in general it can be said that warm rollability was not affected by the temperature of rolling. The prior hot rolling history also had no significant effect on warm rollability.

Several sheets were warm rolled transverse to the direction of hot rolling. Again similar rolling characteristics were observed for all rolling temperatures. The maximum total reductions of 40 percent were accomplished without edge cracks on all sheets indicating that somewhat higher reductions could be obtained at the selected rolling temperatures. Rapid work hardening tendencies were apparent with increasing reductions suggesting a possible upper limit of 55 - 60 percent reduction. However, due to limited sheet bar material, it was not possible to investigate higher reductions in this study.

Room Temperature Tensile Properties

The objective of the Principle Rolling Studies was to develop a rolling procedure which would produce the optimum room temperature tensile properties and the lowest ductile-to-brittle transition temperature in finished sheet. Previous investigators have shown lower ductile-to-brittle transition temperatures in pure chromium sheet which contained small amounts of work after warm rolling.(1) For this reason it was initially planned to perform tensile tests on as-rolled as well as fully annealed chromium composite sheet material. It was soon discovered, however, that tensile strips cut from as-rolled sheet could not be flattened without cracking. This problem persisted in spite of increased flattening temperatures (as high as 1550° F), and reduced pressing speed for sheet specimens warm rolled 8 and 20 percent at all three temperatures.

This brittle behavior is undergoing further study at this time. Some evidence has been gathered which suggests that the decreased ductility of warm worked material may be due to a strain aging process. Examination of electron micrographs prepared from as-rolled sheet specimens has revealed a fine precipitate (perhaps carbide or nitride) within the chromium grams. This precipitate was still apparent in the structure after a 1/2 hour annealing treatment at 1600° F although it was dimished somewhat. The amount of precipitate was found to be further reduced after annealing at 1700 and 1800° F. If the strain aging phenomena is confirmed, further investigations of heat treatment and "fixing" element additions should lead to improved ductility in worked material.

Satisfactory tensile specimens were prepared from warm rolled sheets vacuum annealed at 2000° F for 1/2 hour. Flattening of each individual tensile strip was adequately performed at 1200° F. All specimens were re-annealed for 1/2 hour at 2000° F following the flattening operation. The resulting tensile test data are presented in Table 11.

It can be seen that the surface condition of the test bars played an important part in determining tensile elongation. Without exception, the electropolished specimens were superior to the as-rolled specimens. In addition, both longitudinal and transverse tensile properties appeared dependent upon total final reduction.

Sheets with 40 percent reduction produced consistently higher values of elongation

[1]References cited are listed at the end of this report.

Table 11. Effect of Warm Rolling Variables on the Room Temperature Tensile Properties of Recrystallized Sheet (a)

Sheets(s)	Final Rolling Temperature, F	Final Reduction Percent	Final Rolling Direction(b)	Type Of Surface(c)	Longitudinal Properties: Ultimate Strength, 1000 PSI	Longitudinal: Yield Strength(d) 1000 PSI	Longitudinal: Elongation, Percent	Longitudinal: No. of Tests	Transverse Properties: Ultimate Strength, 1000 PSI	Transverse: Yield Strength(d) 1000 PSI	Transverse: Elongation, Percent	Transverse: No. of Tests
117 & 118	600	40	P	AR	47.8	28.8	7.8	1	41.1	26.6	3.8	2
117	600	40	P	EP	-	-	-	-	45.9	28.0	9.3	3
137	600	40	T	AR	50.3	31.1	9.0	2	45.8	27.7	5.7	2
119	900	40	P	AR	50.6	28.8	8.7	2	49.7	29.6	9.7	2
120	900	40	P	EP	51.0	28.9	12.2	3	48.4	28.0	13.5	3
141	900	40	T	AR	52.4	30.1	10.8	2	47.6	26.0	8.2	2
115 & 116	1200	40	P	AR	51.5	30.6	10.3	1	37.3	27.4	1.5	3
115	1200	40	P	EP	-	-	-	-	48.5	28.0	11.0	1
139	1200	40	T	AR	51.2	30.2	7.0	1	-	-	-	-
133	900(e)	40	P	AR	54.1	34.4	10.0	2	43.5	31.0	3.0	3
133	900(e)	40	P	EP	52.1	29.8	13.5	3	-	-	-	-
134	900(e)	40	T	AR	52.3	30.4	10.0	3	-	-	-	-
121 & 122	600	20	P	AR	35.3	26.5	1.4	5	-	-	-	-
122	600	20	P	EP	48.1	29.9	10.7	2	-	-	-	-
136	600	20	T	AR	40.8	27.5	3.8	3	-	-	-	-
125 & 126	900	20	P	AR	39.6	26.8	2.5	9	35.8	25.5	1.9	2
140	900	20	T	AR	45.1	28.5	5.5	3	-	-	-	-
124 & 138	1200	20	P	AR	45.4	28.7	3.5	3	42.3	26.4	3.8	2
138	1200	20	P	EP	49.5	27.3	12.6	2	-	-	-	-
132	900(e)	20	P	AR	41.2	30.0	2.0	3	-	-	-	-

(a) All sheets annealed 1 hour at 2000°F after warm rolling.

(b) P - Parallel to direction of hot rolling; T - Transverse to direction of hot rolling.

(c) EP - Electro polished; AR - As rolled.

(d) 0.2% offset value.

(e) Hot rolled at 2200°F with a 50 percent intermediate reduction at 1800°F. All others rolled at 2200°F exclusively.

when as-rolled surfaces were evaluated. The results of four tests made with electropolished specimens having 20 percent total reduction were equivalent, however, to the data collected from specimens having 40 percent reduction. Slightly lower yield and ultimate strengths were obtained from transverse specimens.

Rolling temperature had no significant effect on the longitudinal or transverse tensile properties. Similarly, variations in final rolling direction and prior hot rolling history had no apparent effect on tensile test results.

Using data collected during previous rolling trials, together with the room temperature tensile properties discussed here, as a guide, the following warm rolling procedure was selected as the optimum for producing the lowest ductile-to-brittle transition in finished sheet:

Warm Rolling Temperature	-	900° F
Total Warm Work	-	40 Percent
Reduction/Pass	-	7 - 10 Percent
Final Rolling Direction	-	Parallel to Hot Rolling
Final Surface Finish	-	Electropolished

Final annealing treatment for warm rolled sheet was found to influence tensile properties. This investigation is discussed in the section which follows.

SPECIAL ROLLING STUDIES

Optimum rolling procedures developed during the previously described rolling studies were used to produce sheet samples for investigation of recrystallization and tensile behavior with variations in annealing treatment. The data pertinent to these experimental rolling trials are in Table 24 in the Appendix.

Recrystallization and Softening Behavior

The recrystallization behavior of warm rolled sheet was determined for total reductions of 10, 20, 40, 60 and 70 percent. Hot rolling reductions were adjusted to provide a finished warm rolled sheet thickness of 0.050 inches for each of these reductions. Samples from finished sheets were vacuum annealed for 1/2 hour and one hour at temperatures from 1600 to 2000° F. The annealed samples were then mounted, polished and etched for determination of structure and hardness.

The recrystallization temperatures and hardnesses of the as-rolled and annealed samples are given in Table 12 and Figures 64 and 65. The as-rolled hardnesses increased progressively with increasing reduction indicating a relatively uniform rate of work hardening. Increasing the 1/2 hour annealing temperature produced a rapid decrease in hardness up to 1700° F for samples with 40, 60 and 70 percent warm work. For samples with 10 - 20 percent work, the hardness decrease was more gradual reaching a minimum at 2000° F.

An additional 1/2 hour annealing time had little effect with the exception of the specimens warm worked 70 percent. In this case, near minimum hardness was realized at 1600° F with the longer annealing time. However, examination of the microstructure did not confirm full recrystallization at this temperature.

The hardness minima for the higher reductions occurred at those 1/2 hour annealing temperatures for which fully recrystallized structures were obtained. The photomicrographs in Figures 66 through 68 show the structural changes which resulted from the 1/2 hour anneals at various temperatures for specimens reduced 40, 60 and 70 percent. The structures observed for the 10 and 20 percent reductions failed to show the effects of the small amount of work even in areas close to the as-rolled surface. For specimens with 40 percent and greater warm work, partial recrystallization was observed after 1/2 hour at 1600° F with recrystallization being complete after 1/2 hour at 1700° F.

Heat Treatment Investigation

The observed recrystallization behavior was used as a guide in the selection of annealing temperatures which would potentially enhance the room temperature tensile properties of sheet with 40 percent warm work. Six sheets were rolled to provide as-rolled, stress relieved, and recrystallized tensile specimens. A temperature of 1600° F was selected for stress relieving, and temperatures of 1700 to 2000° F were used to provide annealed specimens.

Difficulties were again experienced in the flattening of as-rolled and stress relieved tensile strips. As-rolled specimens were finally taken from an exceptionally flat sheet

Table 12. Recrystallization Behavior of 50 Mil Sheet Finish-Rolled at 900° F

Sheet	Final Reduction, Percent	Annealing Time, Hours	Knoop Hardness, 25 Gram Load(a)				
			As Rolled	Hydrogen Annealed at Indicated Temperature(b)			
				1500 F	1700 F	1800 F	1900 F
163	8.8	½	206	236	209	180	163
		1	-	252	235	206	194
164	18.8	½	212	224	204	173	171
		1	-	260	209	181	184
165	40.0	½	220	271	<u>180</u>	180	180
		1	-	231	<u>191</u>	211	181
166	61.0	½	252	250	<u>175</u>	171	175
		1	-	247	<u>197</u>	180	179
167	70.0	½	268	213	<u>170</u>	164	173
		1	-	198	<u>195</u>	188	186

(a) Average of six readings for each condition.

(b) Underlined values indicate temperature where recrystallization is complete as determined by microscopic examination.

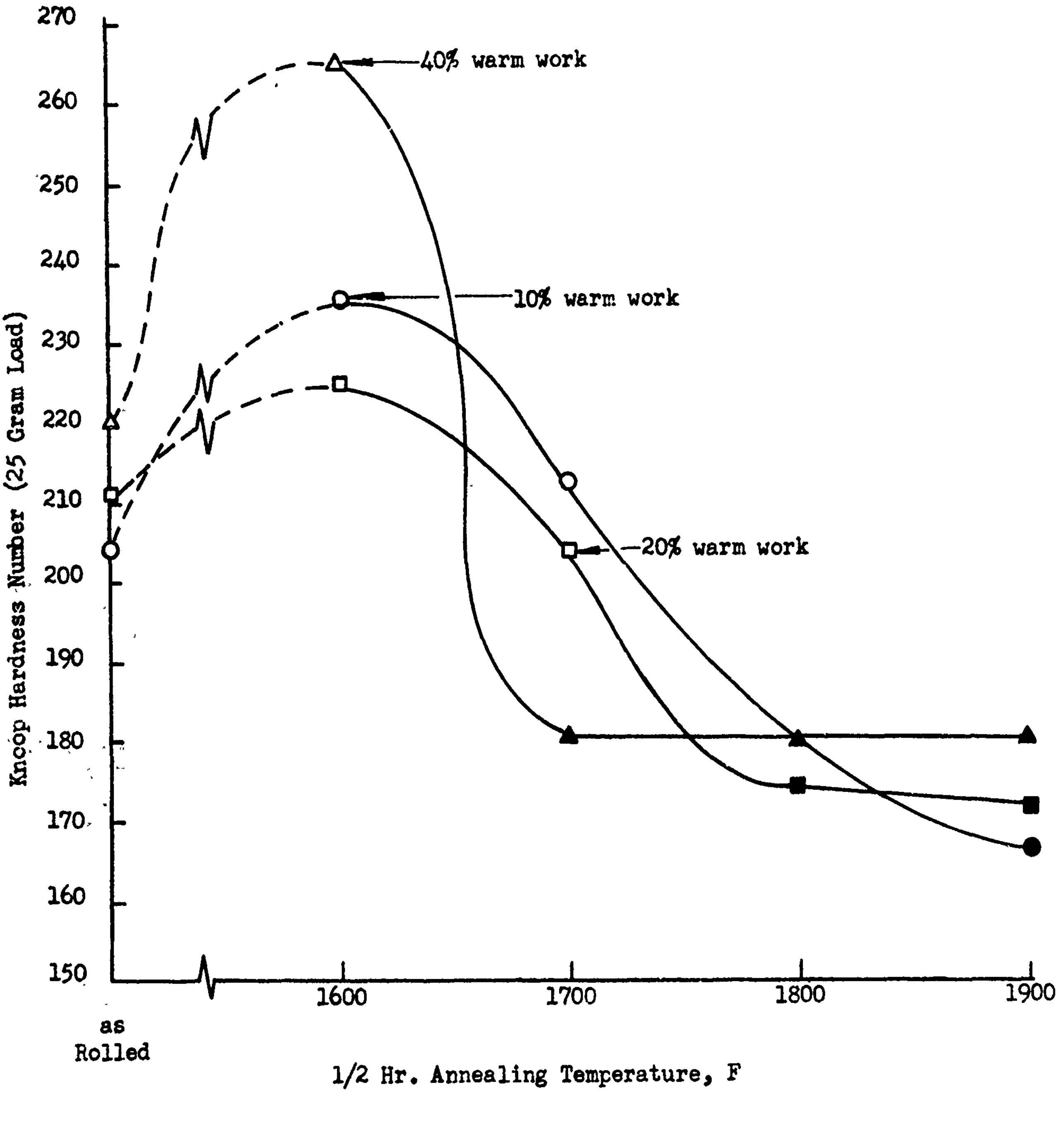


Figure 64 - Effect of Annealing Temperature on the Room Temperature Hardness of 50 Mil Sheet Finish Rolled @900 F

Solid Symbols Indicate Complete Recrystallization

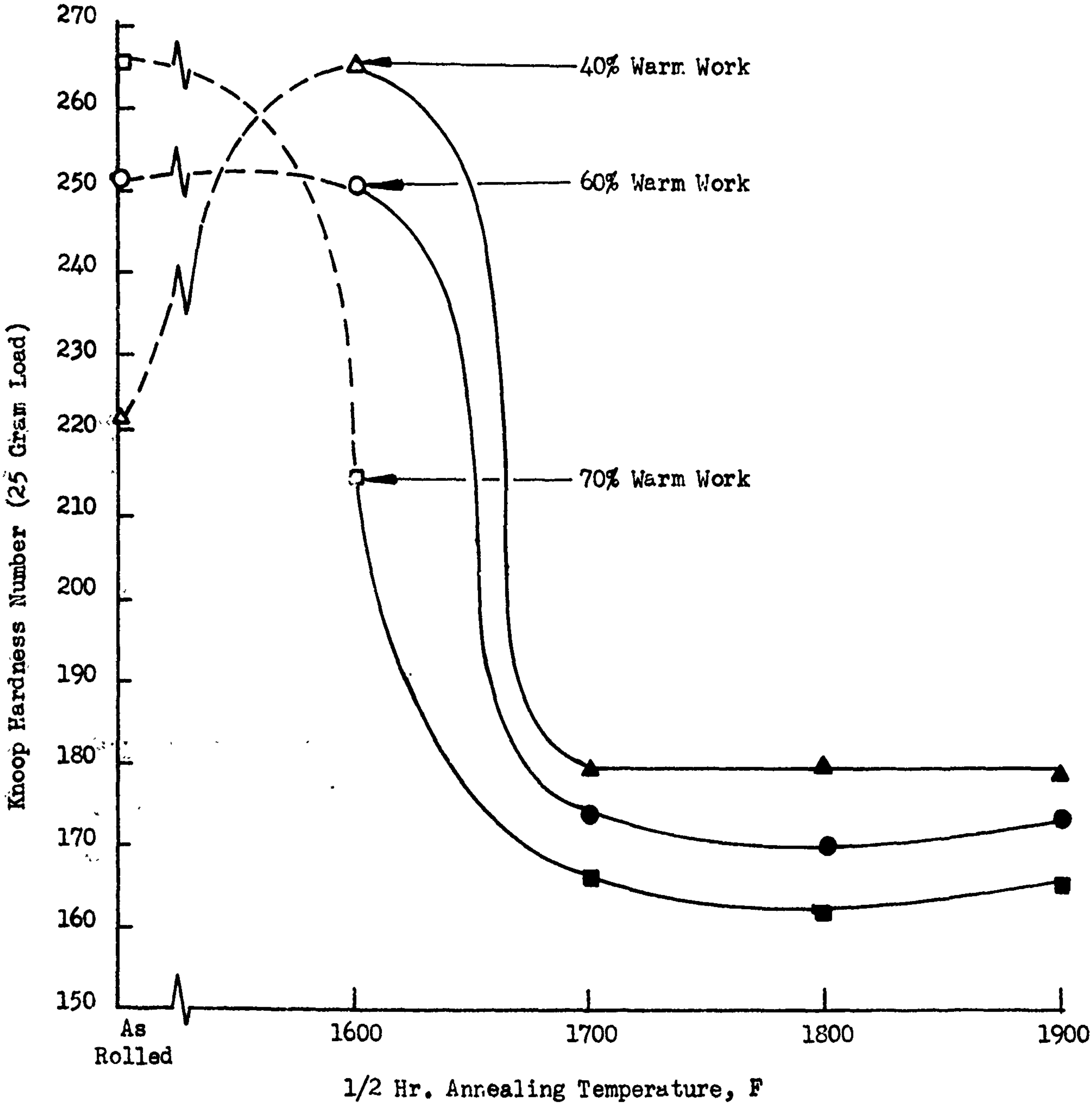
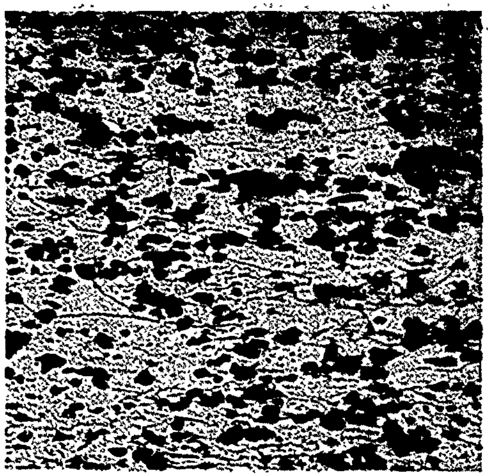
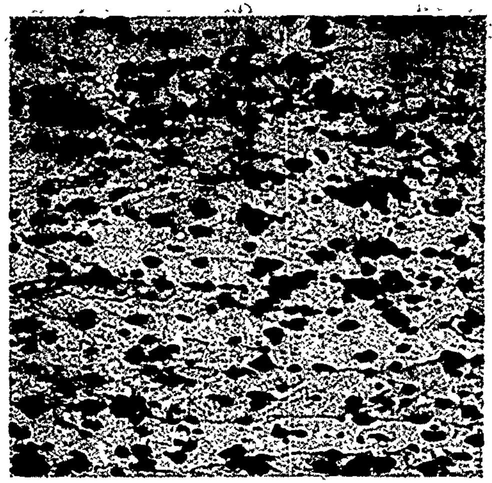


Figure 65 - Effect of Annealing Temperature on the Room Temperature Hardness of 50 Mil Sheet Finish Rolled @900 F

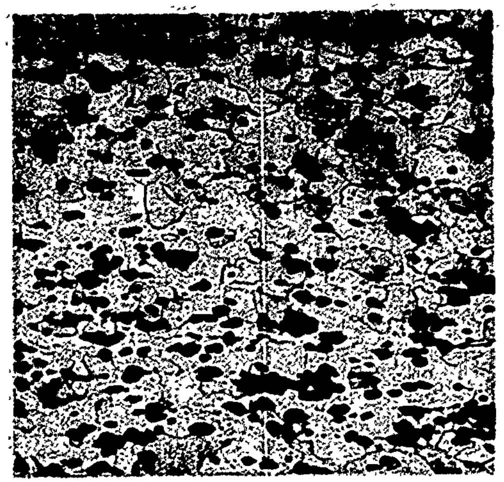
Solid Symbols Indicate Complete Recrystallization



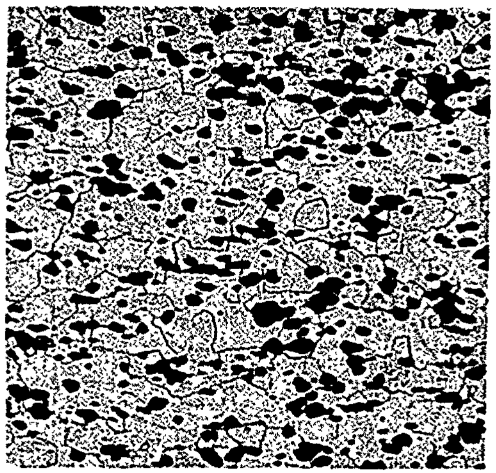
250X KHN - 220 P-178
As Rolled



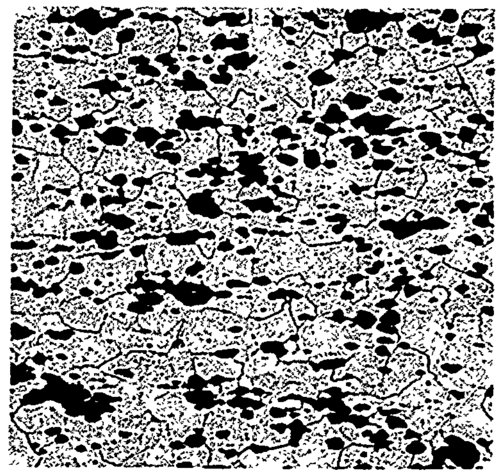
250X KHN - 271 P-178
½ Hour @1600 F



250X KHN - 180 P-178
½ Hour @1700 F

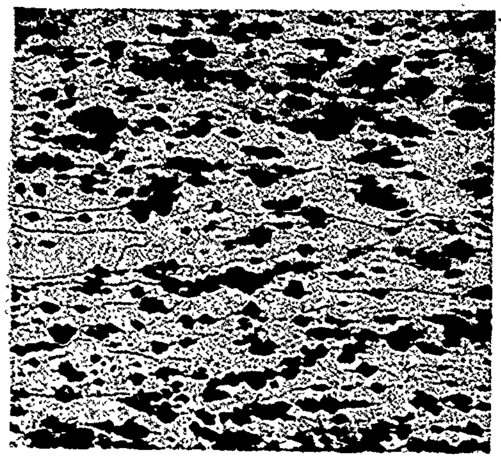


250X KHN - 180 P-178
½ Hour @1800 F

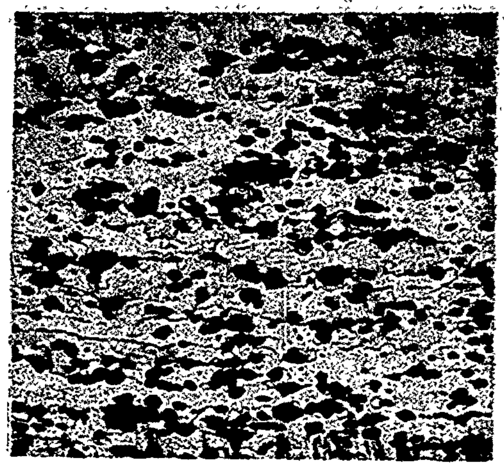


250X KHN - 180 P-178
½ Hour @1900 F

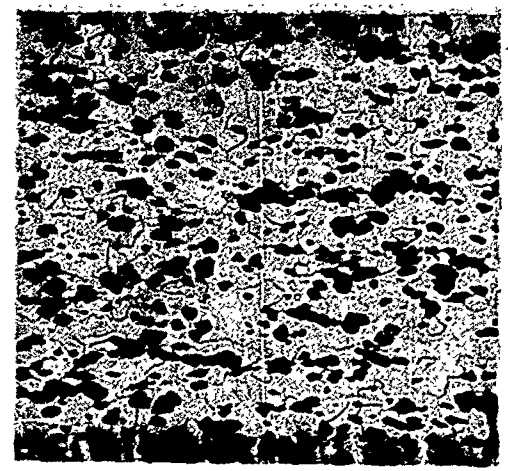
Figure 66 - Recrystallization Behavior of 50 Mil Sheet Finish Rolled @900 F With 40 Percent Warm Work



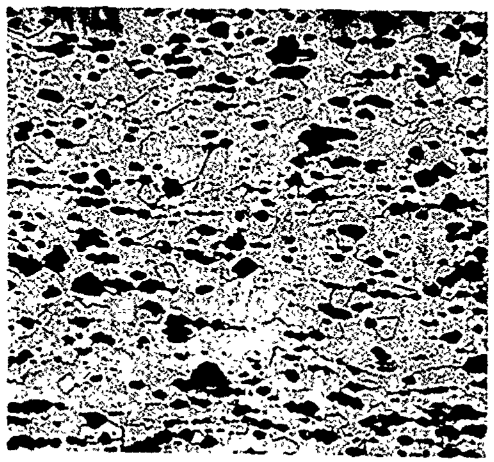
250X KHN - 252 P-179
As Rolled



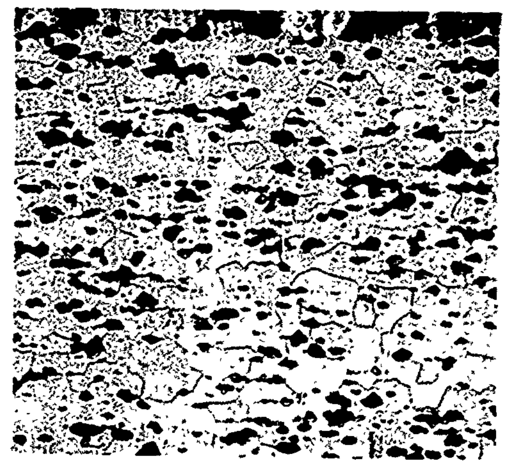
250X KHN - 250 P-179
½ Hour @1600 F



250X KHN - 175 P-179
½ Hour @1700 F

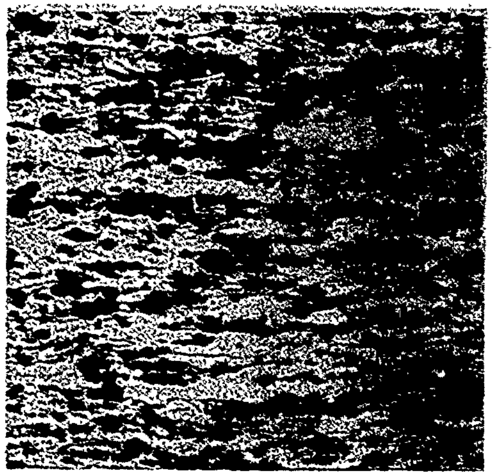


250X KHN - 171 P-179
½ Hour @1800 F

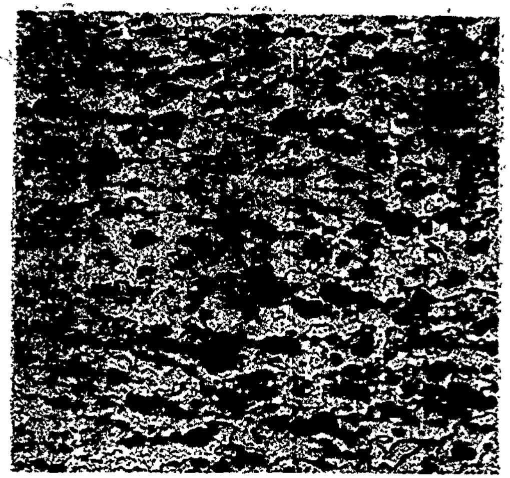


250X KHN - 175 P-175
½ Hour @1900 F

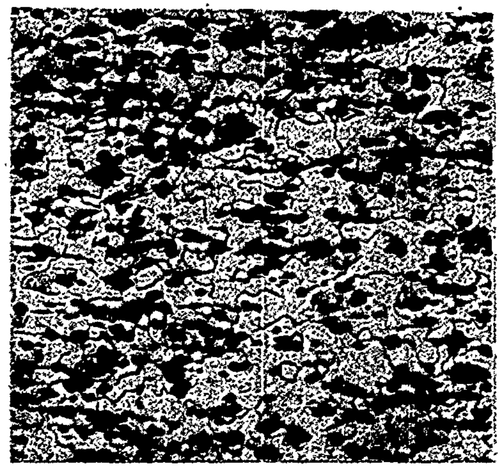
Figure 67 - Recrystallization Behavior of 50 Mil Sheet Finish Rolled @900 F With 60 Percent Warm Work



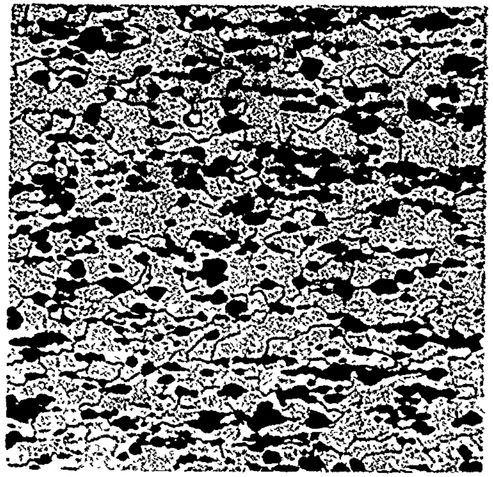
250X KHN - 268 P-182
As Rolled



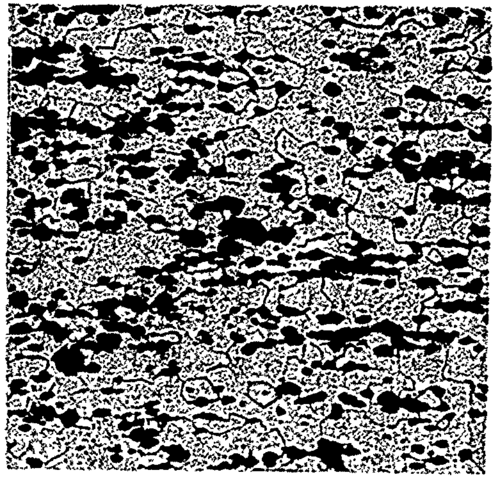
250X KHN - 213 P-182
½ Hour @1600 F



250X KHN - 170 P-182
½ Hour @1700 F



250X KHN - 164 P-182
½ Hour @1800 F



250X KHN - 173 P-182
½ Hour @1900 F

Figure 68 - Recrystallization Behavior of 50 Mil Sheet Finish Rolled @900 F With 70 Percent Warm Work

to avoid the necessity of flattening. Specimens heat treated at 1600° F were successfully flattened by heating to 1500° F. All heat treatments were performed in vacuum for a total of one hour at the selected temperature (1/2 hour prior to flattening and 1/2 hour after flattening). Tensile tests were conducted according to previously described procedures using specimens with electropolished surfaces.

Room Temperature Tensile Test Results

The results of individual tensile tests given in Table 13 show the effect of variations in annealing temperature. The longitudinal tensile property data have been averaged and are presented in Figure 69. As-rolled specimens displayed brittle behavior, as expected, with no apparent yield and high values of ultimate strength. Two of the three bars which were stress relieved at 1600° F gave good elongation in the longitudinal direction. The third bar displayed a much higher yield strength and failed in a near brittle manner outside of the gage length. The one transverse bar also failed in a brittle manner. This marginal ductility behavior could be explained by the suspected strain aging effect which is not completely removed by annealing at 1600° F. The consistently high values of elongation obtained from specimens annealed at 1700 and 1800° F are in agreement with electron micrographs which indicated a re-solution of precipitate at these annealing temperatures.

The recrystallized specimens generally produced consistent test results with the exception of the two test bars annealed at 1700° F which failed prematurely at hole locations. Transverse tensile properties of the recrystallized specimens were similar to the longitudinal properties indicating only minor directional characteristics. The maximum average room temperature elongation was realized from both longitudinal and transverse specimens annealed at 1800° F. Values of yield and ultimate strength decreased with increasing annealing temperature.

The lower elongations observed for specimens annealed at 2000° F is in disagreement with the lower yield strengths shown. There were no microstructural changes apparent with increasing annealing temperature at normal optical magnifications. Studies of electron micrographs should be made to determine any subgrain phenomena which might contribute to an explanation of this anomaly.

An annealing temperature of 1800° F was selected for the optimum sheet studies discussed in the following section.

Table 13. Effect of Annealing Temperature on the Room Temperature Tensile Properties 50 Mil Sheet[a]

Sheet	Annealing Temperature,[b] F	Longitudinal Properties: Ultimate Strength, 1000 PSI	Longitudinal Properties: Yield Strength,[c] 1000 PSI	Longitudinal Properties: Elongation in 1 Inch,[d] Percent	Transverse Properties: Ultimate Strength, 1000 PSI	Transverse Properties: Yield Strength,[c] 1000 PSI	Transverse Properties: Elongation in 1 Inch,[d] Percent
169	As Rolled	73.0	(f)	0	-	-	-
		71.3	(f)	0	-	-	-
		55.0[e]	(f)	0	-	-	-
211	1600	53.3	36.1	14.4	47.5	(f)	0
		55.3	39.6	16.5	-	-	-
		50.2	46.0-43.5	1.8[e]	-	-	-
209	1700	53.6	36.5-31.6	19.0	46.2	37.9-33.1	3.7[e]
		53.8	36.2-32.8	19.0	51.5	39.2-33.0	15.7
		39.0	35.4-32.8	0.8[e]	-	-	-
208	1800	53.0	32.2-31.2	19.0	50.3	33.1-28.8	18.5
		52.6	31.6	20.0	50.5	33.2-27.2	16.0
		55.7	34.0-31.8	18.5	-	-	-
203	1900	52.9	29.1	20.0	50.3	27.3-26.4	15.5
		53.0	29.6	18.2	49.6	28.2	9.2[e]
		51.9	28.4-27.2	15.5	50.0	27.2-26.2	13.0
210	2000	50.6	28.4-27.0	14.0	46.5	27.5	6.6
		49.7	27.9	10.0	47.2	25.8	8.7
		54.0	29.8	12.5	-	-	-

(a) All sheets finish-rolled 40% at 900 F.
(b) Specimens vacuum annealed for ½ hour, flattened at 1500 F in air, and re-annealed for ½ hour.
(c) Upper and lower yield reported where observed. Others 0.2% offset.
(d) Specimens electropolished prior to test.
(e) Specimens failed outside of gage marks.
(f) No yield point observed.

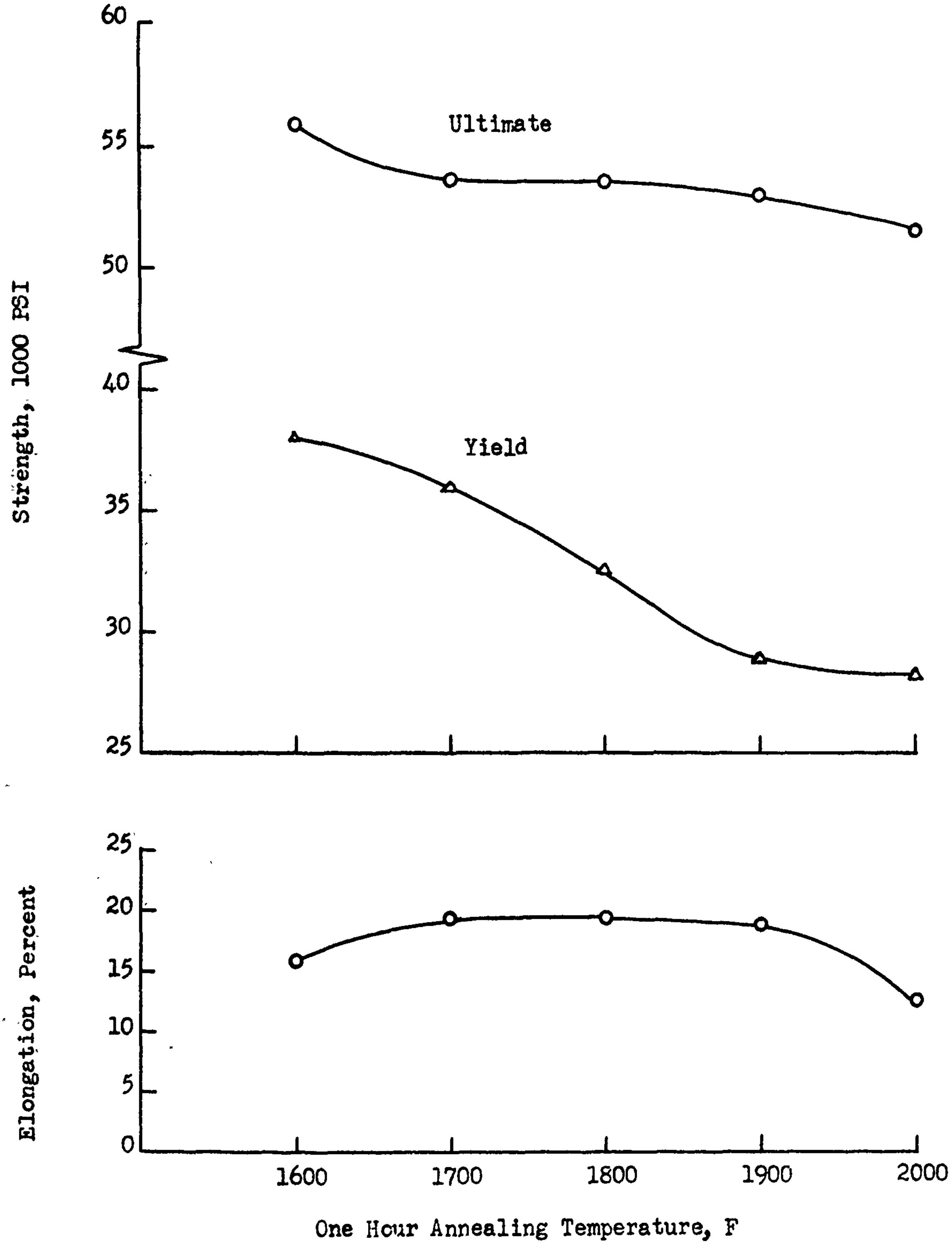


Figure 69 - Effect of Annealing Temperature on the Room Temperature Tensile Properties of 50 Mil Sheet

OPTIMUM SHEET STUDIES

The extruded Chrome-30 composite used throughout this program is known to possess several outstanding properties which would be desirable in a sheet product. These include: (1) a ductile-to-brittle transition below room temperature; (2) a high degree of oxidation and scaling resistance; (3) a unique resistance to erosion; (4) a resistance to nitridation; and (5) a useable high temperature strength. A total of 20 sheets were processed by optimum procedures developed in this program to provide material for a study of these properties. Rolling data for these sheets are included in Table 24 in the Appendix and a discussion of the testing program follows.

Ductile-to-Brittle Transition Behavior

Tensile specimens from three optimum sheets were annealed at 1800° F and tested at temperatures ranging from 10 to 100° F. The results of the individual tensile tests are given in Table 14 and Figure 70. The transition temperature from ductile-to-brittle behavior was estimated as the temperature at which the elongation at fracture decreased to 1/2 of the maximum value, or approximately 45° F for the longitudinal sheet specimens tested. This transition temperature is somewhat higher than that previously observed in extruded material (10° F, as shown in Figure 22), possibly due to differences in specimen configuration and surface preparation. Very little change was noted in ultimate tensile and yield strength with decreasing temperatures.

This transition temperature is unusually low for recrystallized chromium. Pure iodized chromium, for example, is known to have a ductile-to-brittle transition near 750° F in the recrystallized condition. The factors responsible for this improvement in ductile behavior have not been definitely determined as yet although there is considerable strength in the argument that the dispersed magnesium oxide acts as a scavenging agent for detrimental interstitial elements.

Additional tests were made on recrystallized sheet material with 55 percent prior warm work (sheet 206). Results from one test at each temperature were nearly identical to those shown in Table 14, indicating that the amount of warm rolling prior to annealing has no significant effect on low temperature strength or ductility.

Several tensile specimens from an as-rolled sheet with 7.9 percent work (sheet 173) were tested to determine the temperature at which ductile behavior could be realized. Brittle failures were obtained at all temperatures to 1000° F for this lightly worked material which is in agreement with previous observations regarding the brittle nature of as-rolled sheet.

Oxidation and Nitridation Behavior

Rectangular specimens from sheet 201 were oxidized for 24 hour periods at 1800, 2200 and 2400° F. The results are listed in Table 15. None of the specimens showed evidence of oxide spalling or blistering as a result of the oxidation exposure and rapid cool on removal from the furnace. However, the oxide layer formed on the 2400° F specimens could be separated whereas those formed at lower temperatures were well bonded to the base metal. The average oxidation rate curves for each temperature are

Table 14. Tensile Properties as a Function of Temperature for Recrystallized Fifty Mil Sheet[(a)]

Sheet Specimen Number	Test Temperature, F	Ultimate Strength, 1000 PSI	Yield Strength,[(b)] 1000 PSI	Elongation in 1 Inch,[(c)] Percent
212-2	10	51.0	(d)	0
212-1	30	45.4	(d)	0
212-6	40	56.7	38.0	8.5
213-4	40	52.8	38.4-36.3	6.2
213-5	40	54.0	37.9-36.7	7.2
212-3	50	56.8	35.5	12.5
212-4	50	57.0	38.6	11.0
213-3	50	52.7	35.6-33.5	10.0
212-5	60	55.7	39.3	13.2
212-7	60	56.3	35.7	14.0
213-2	60	54.0	35.3	14.5
208-1	75	53.0	32.2-31.2	19.0
208-2	75	52.6	31.6	20.0
208-3	75	55.7	34.0-31.8	18.5
213-1	100	51.0	30.8	18.5

(a) Warm rolled 40% at 900 F and annealed 1 hour at 1800 F.

(b) Upper and lower yield reported where observed. Others 0.2% offset.

(c) Specimens electropolished prior to test.

(d) No yield point observed.

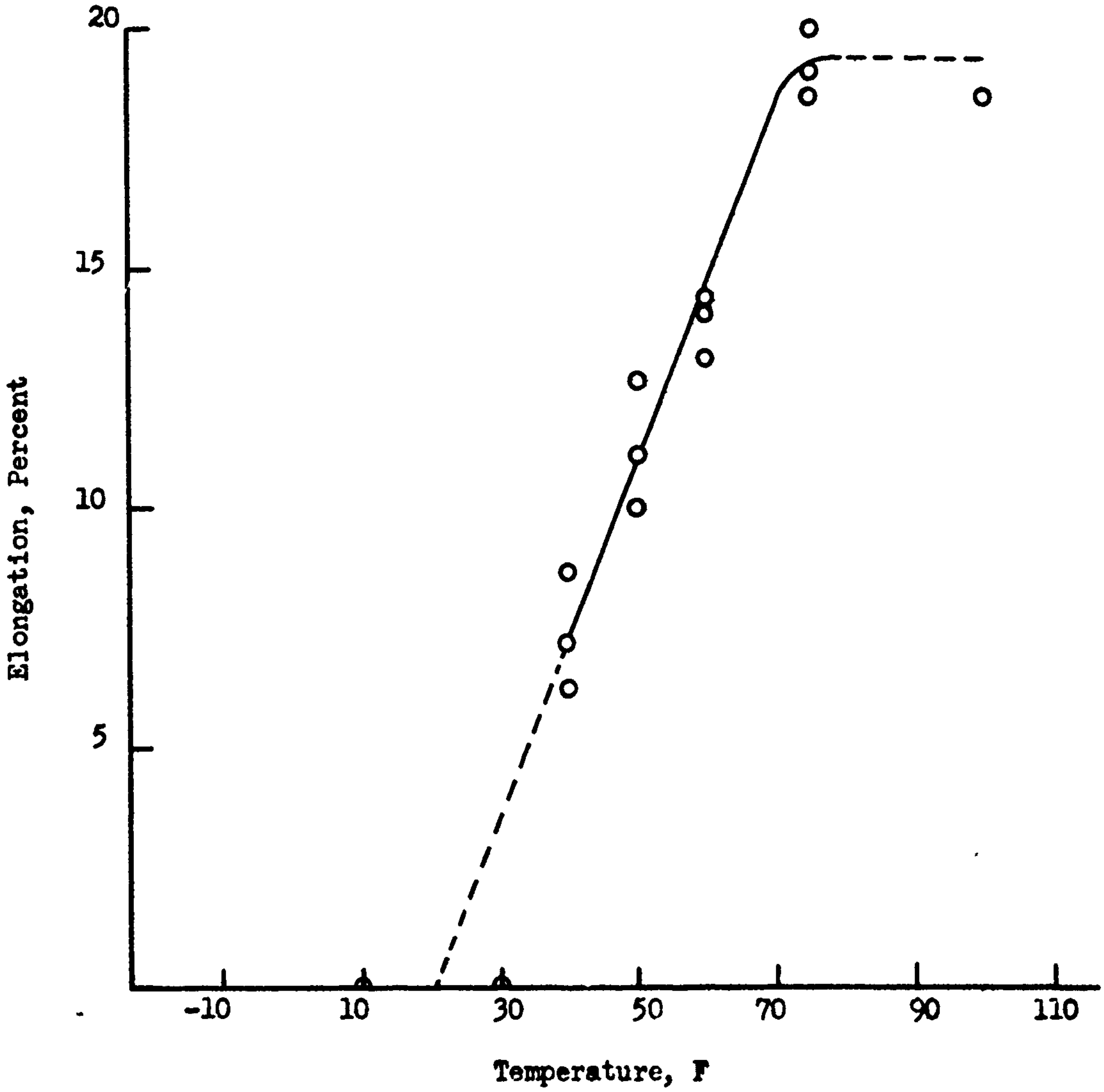


Figure 70 - Ductile-to-Brittle Tensile Transition for Recrystallized 50 Mil Sheet

Table 15. Oxidation Behavior of Chrome-30 Sheet Specimens[a]

Exposure Temperature, F	Trial Number	Total Weight Gain, mg	Weight Gain, mg/cm^2	Average Depth of Nitride Layer, Mils
1800	1	5.3	1.44	0
1800	2	3.4	.93	0
1800	3	6.3	1.72	0
2200	1	14.9	3.98	0
2200	2	13.6	3.63	0
2200	3	16.8	4.59	0
2400	1	78.7	21.55	4.5
2400	2	82.0	21.63	3.8
2400	3	95.3	25.10	4.6

(a) Twenty-four hour exposure in dry air flowing at 1 SCFH.

shown in Figure 71 and a typical 2400° F test specimen is shown in Figure 72. The 2200 and 2400° F data represent a significant improvement over data published for unalloyed chromium. This improvement is believed to be due to the formation of an oxide layer which consists primarily of magnesio chromite spinel ($MgCr_2O_4$). The thermal properties of this spinel are known to be more compatible with those of the chromium substrate in comparison to the normal Cr_2O_3 oxidation product. The lower thermal strains resulting from a closer match would allow diffusion controlled oxidation to be maintained to higher temperatures. The relatively dense, crack-free oxide coatings observed on the 2200 and 2400° F specimens tend to support this premise.

It is believed that this spinel is also responsible for retarding nitrogen diffusion to the Chrome-30 interface. Each oxidation test specimen was sectioned, polished and examined after testing to determine the extent of nitrogen penetration. A nitride layer was detected on all of the 2400° F samples extending to an average depth of 4.3 mils. There was no evidence of nitride formation on the lower temperature samples. Tests conducted in an identical manner on extruded unalloyed chromium prior to this study revealed nitrogen pick-up at 2200° F as well as 2400° F with a nitride penetration extending throughout the entire cross-section on the 2400° F sample. The photomicrograph in Figure 73 shows a typical Chrome-30 specimen after a 24 hour oxidation at 2400° F.

Although Chrome-30 sheet material is not completely insensitive to nitridation, the results indicate a significant improvement over unalloyed chromium. The addition of rare earth alloying elements to chromium composites should produce a material which is highly resistant to nitridation.

Erosion Behavior

A brief experiment was conducted to determine the resistance of Chrome-30 sheet to the erosive flow of high velocity-high temperature gases. Extensive studies have been made in the past using a kerosene oxygen torch to provide an oxygen rich gas stream capable of penetrating a 1/2 inch thick sample of molybdenum, chromium or tungsten in less than two minutes at a heat flux of 400 BTU/ft^2/second. Extruded Chrome-30 sample have been exposed under identical conditions for 15 minutes without damage even though surface temperatures above the melting point of chromium were recorded.[2]

This standard test procedure was used to evaluate a curved sample of Chrome-30 sheet. A test sample, 1-1/4 inches wide by 5-1/2 inches long from sheet 212, was heated to 1200° F and bent over a 3/8 inch die radius to an included angle of 40 degrees. The sample was then fitted to a contoured fire brick placed at 5-1/4 inches from the nozzle of the kerosene oxygen torch. The sample was given an initial five minute exposure using a mixture of 32 pounds per hour of kerosene and 1260 scfh of oxygen which produced a stabilized corrected optical surface temperature of 3060° F. The test was then continued for 5 additional minutes at progressively increased fuel settings. The final mixture, 34 pounds per hour of kerosene and 1230 scfh of oxygen with a flame temperature of approximately 6000° F, produced a stabilized temperature of 3350° F on the curved specimen surface.

The tested sample is shown in Figure 74. An adherent, crack-free oxide formed on the exposed surface producing a thickness increase of 0.004 inches which is typical

[2] References cited are listed at the end of this report.

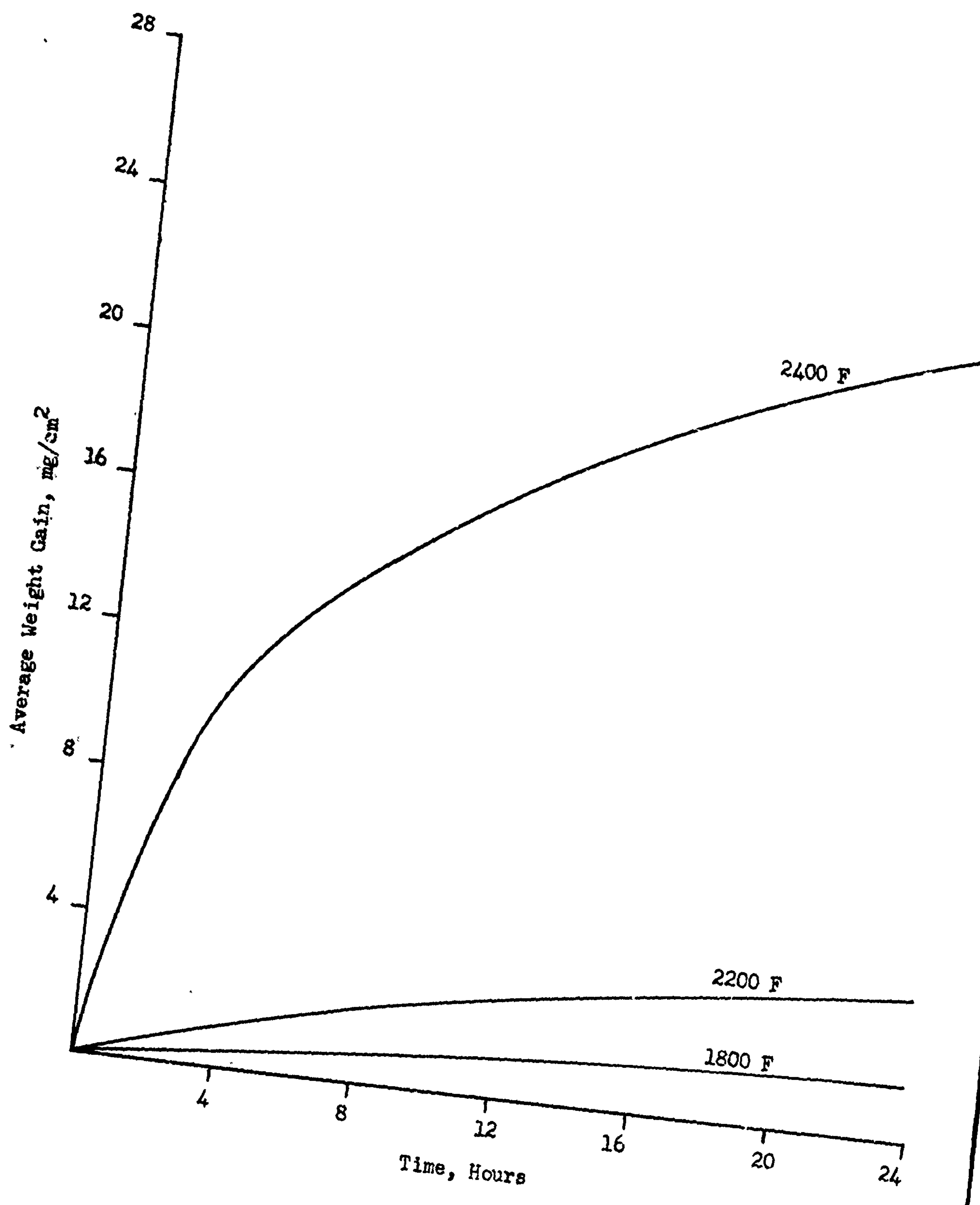


Figure 71 - Effect of Temperature on the Oxidation Rate of Recrystallized 50 Mil Sheet

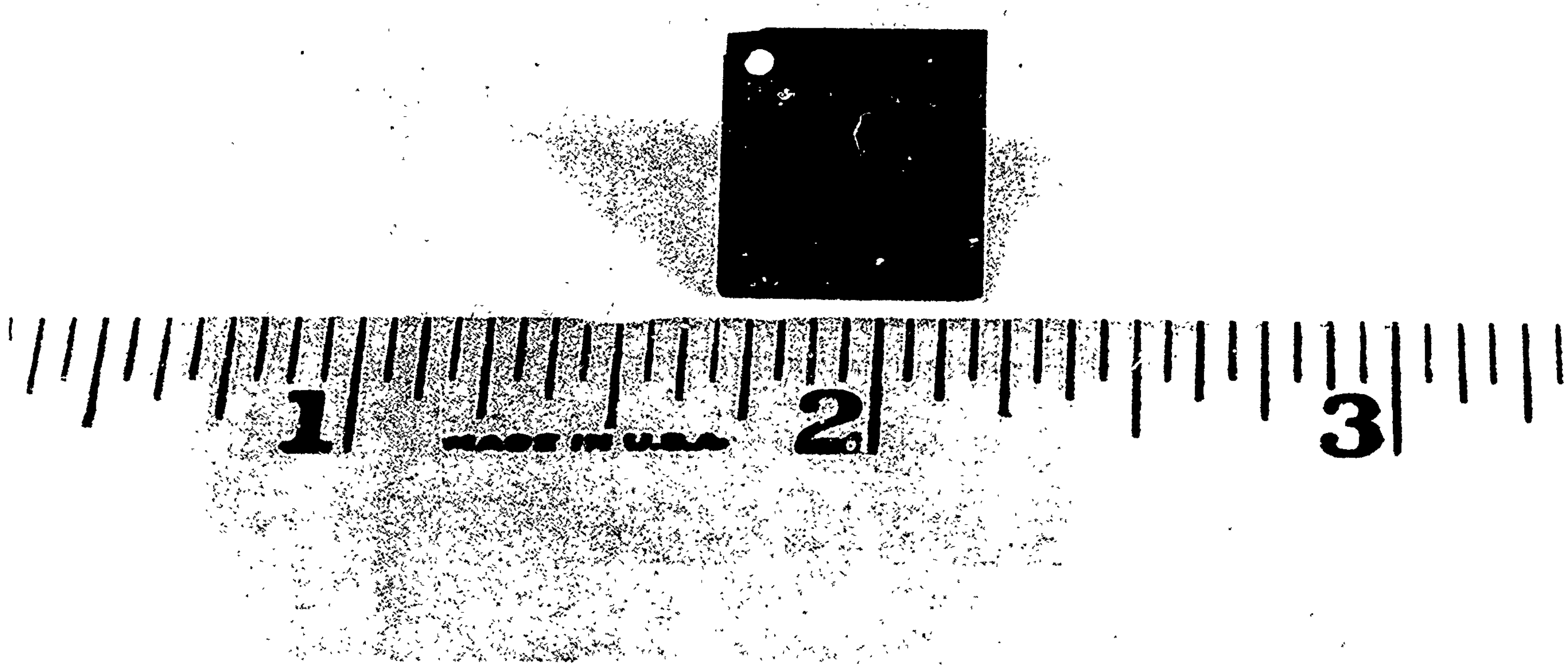


Figure 72 - Sheet Sample After 24 Hour Oxidation @2400 F

Figure 73 - Nitride Layer Formed During 2400 F Oxidation

under the conditions of this test. The thermal properties of this coating are undoubtedly responsible for the outstanding erosion resistance demonstrated in this and similar tests.

High Temperature Tensile Behavior

The results of short time elevated temperature tensile tests performed on optimum sheet specimens are given in Table 16. These data have been averaged and are graphically illustrated in Figure 75. It can be seen that recrystallized sheet exhibits a moderate decrease in tensile strength with increasing temperature to 1800°F after which a more gradual decrease in strength is apparent with increasing temperature. The yield strength was found to decrease at a slightly slower rate with increasing temperature. These data are in agreement with the elevated temperature tensile properties of extruded Chrome-30 with the exception of the distinctive decrease in elongation which occurred at 2200°F. Examination of the microstructures of these three test specimens failed to reveal the nature of this ductility phenomenon.

Good reproducibility was shown for the three specimens tested at each temperature indicating good uniformity throughout each individual sheet and from one sheet to another.

The elevated temperature strength of recrystallized Chrome-30 sheet is not outstanding as these data show. However, the strength levels are within a useable range for many high temperature applications. The improvement of high temperature strength is a logical step to be taken in the continuing development of chromium composites.

Stress Rupture Behavior

A total of 75 stress rupture specimens were prepared from 14 optimum sheets for testing at the ASD Applications Laboratory. The results of these tests will be published by the Air Force as a supplement to this report.

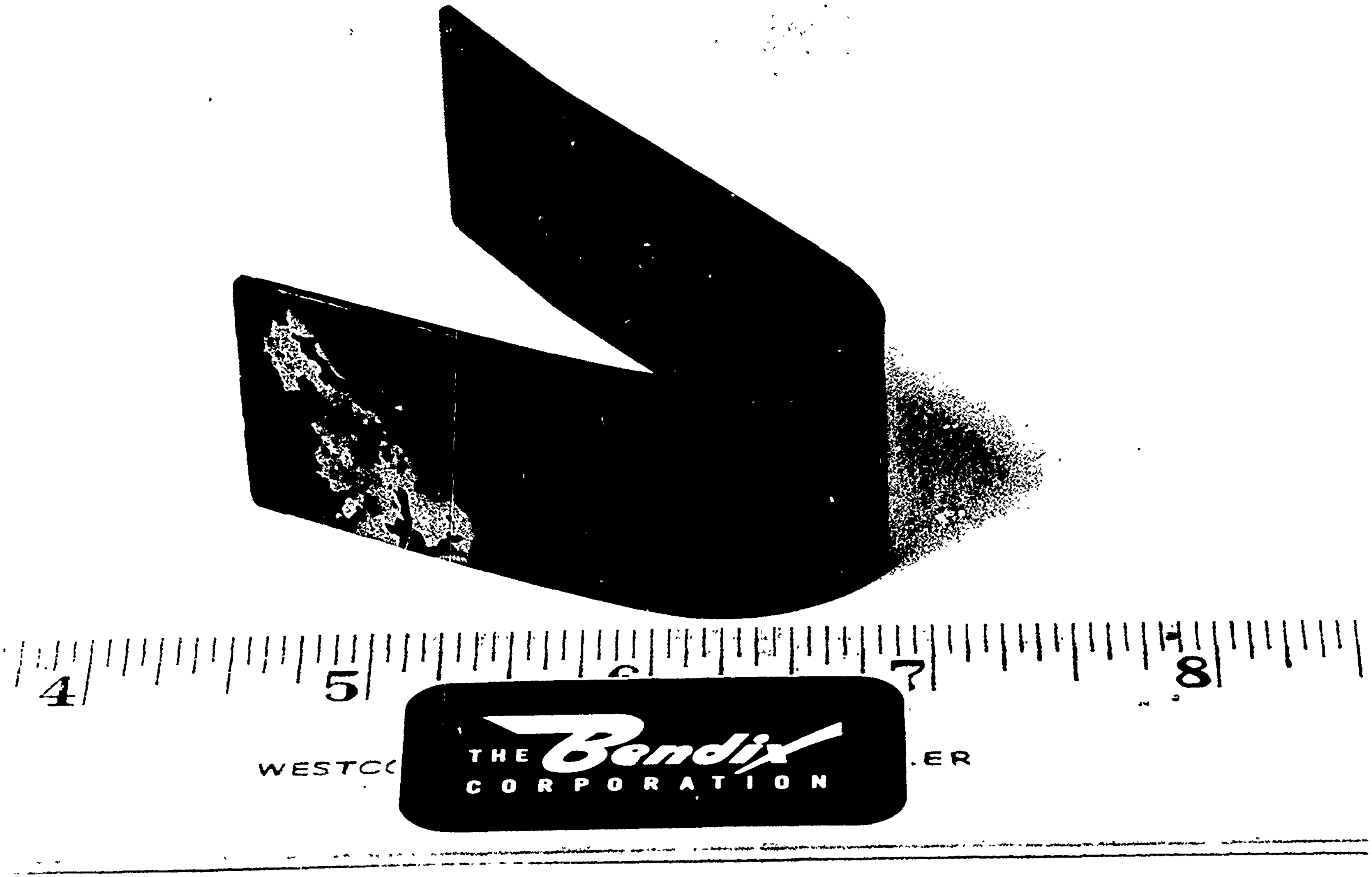


Figure 74 - Chrome-30 Sheet Sample After 10 Minute Erosion Test

Table 16. Elevated Temperature Tensile Properties of Recrystallized 50 Mil Sheet

Sheet Specimen Number	Test Temperature, F	Ultimate Strength, 1000 PSI	0.2% Yield Strength, 1000 PSI	Elongation in $1\frac{1}{4}$ Inches, Percent
202-1	1000	36.9	25.2	16.0
202-2	1000	37.6	25.6	17.5
202-3	1000	37.3	25.0	16.8
202-5	1400	25.2	16.3	25.2
202-6	1400	23.2	16.0	26.0
205-3	1400	27.0	17.3	25.4
200-2	1800	12.0	8.5	58.0
200-4	1800	11.9	8.9	46.4
205-5	1800	11.7	9.5	48.5
205-1	2200	5.2	4.7	26.2
205-6	2200	5.0	4.2	24.0
207-1	2200	4.9	4.4	33.0
207-4	2400	3.0	2.7	57.5
207-5	2400	3.1	2.6	66.5
207-6	2400	3.0	2.6	85.0

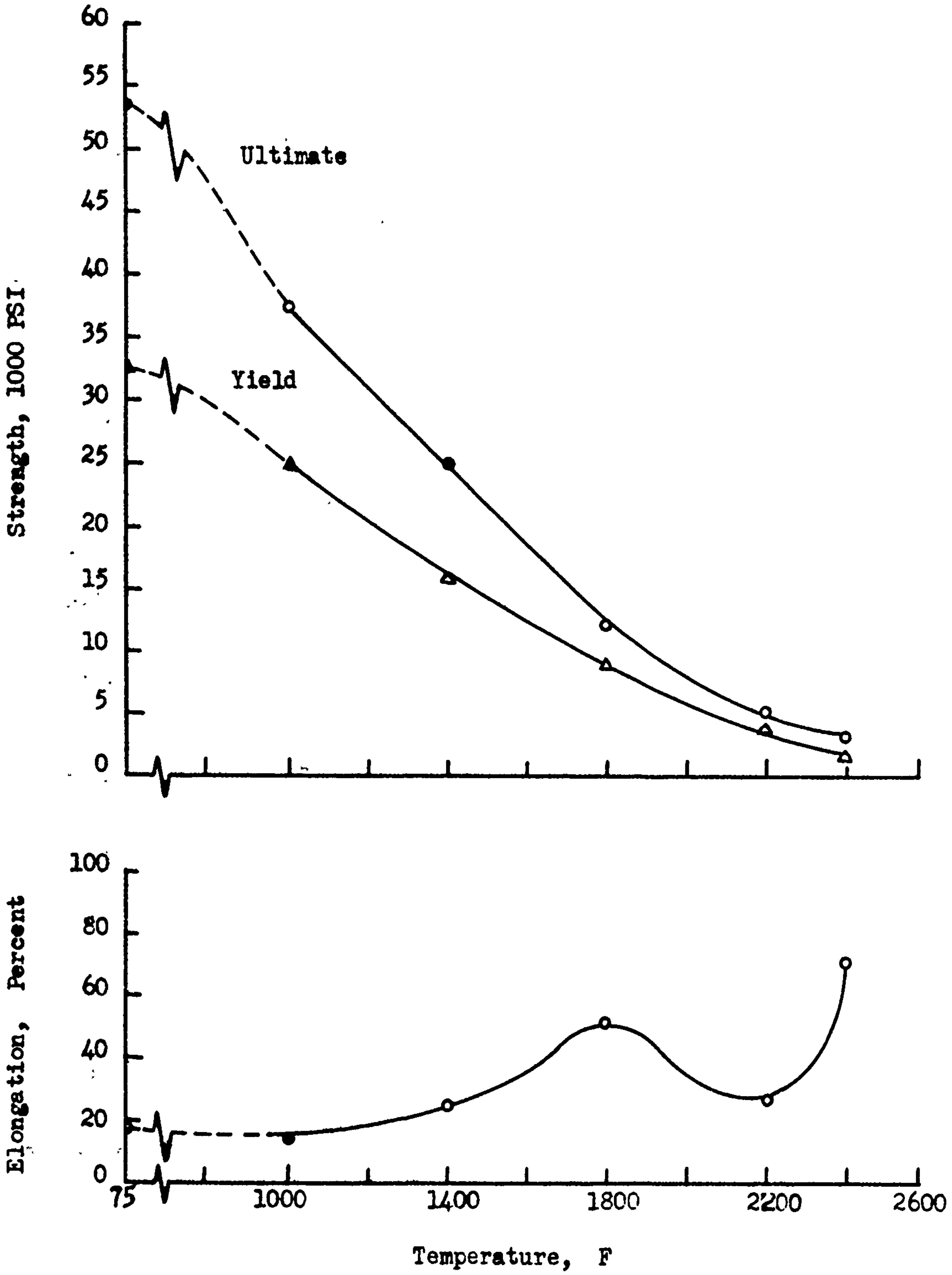


Figure 75 - Effect of Elevated Temperature on the Tensile Properties of Recrystallised 50 Mil Sheet

RECOMMENDATIONS FOR FUTURE WORK

Research studies, sponsored by Bendix at the Bendix Research Laboratories Division, the Bendix Products Aerospace Division and the University of Illinois, are providing fundamental information concerning the mechanisms responsible for the unique behavior of chromium composite systems. The results of this work show that significant improvements in properties can be realized with additional development effort. It is also very apparent that the composite mechanism is applicable to other metallic systems.

These observations together with the findings from this report suggest the following areas of future study:

1. Strengthening by oxide dispersion and solid solution alloying.
2. Suppression of nitrogen diffusion by variation in oxide dispersion and/or rare earth additions.
3. Welding, joining and sheet forming to provide useable structural products.
4. Scale-up rolling development to meet size requirements.

REFERENCES

(1) Yoshida, S., Ohba, Y., and Nagata, N., "Effect of Pre-strain on the Ductility of Pure Chromium", Trans. National Research Institute for Metals, 4 (1) 1962, pp. 22-27.

(2) Masterson, J., "Chromium Composites for Extreme Environmental Conditions", SAE Journal, 4 (6) June 1962, pp. 31-33.

APPENDIX

Table 17. Chrome-30 Extrusion Billet Histories

Billet Number	Billet Length, Inches	Billet Diameter, Inches	Machined Weight, Pounds	Sintered Density, g/cc	Density, As Percent of Theoretical	Ultrasonic Character
432	6.170	2.830	8.85	5.63	84.4	Clear
437	5.965	2.830	8.58	5.62	84.2	"
438	5.930	2.834	8.47	5.65	84.7	"
439	6.005	2.830	8.56	5.57	83.5	"
440	6.016	2.836	8.73	5.65	84.7	"
441	5.985	2.830	8.59	5.60	83.9	"
433	5.450	2.831	7.83	5.64	84.6	"
434	5.788	2.835	8.28	5.58	83.7	"
443	6.032	2.820	8.78	5.61	84.0	"
447	6.015	2.829	8.64	5.68	85.0	"
436	6.025	2.831	8.74	5.67	84.9	"
445	6.004	2.831	8.73	5.60	83.9	2 Substandard indications
446	6.020	2.831	8.62	5.62	84.2	Clear
454	5.266	2.831	7.54	5.61	84.0	"
442	6.016	2.820	8.76	5.63	84.4	"
444	5.985	2.813	8.57	5.59	83.8	"
430	4.775	2.829	6.97	5.69	85.2	"

Table 17. Chrome-30 Extrusion Billet Histories — Continued

Billet Number	Billet Length, Inches	Billet Diameter, Inches	Machined Weight, Pounds	Sintered Density, g/cc	Density, As Percent of Theoretical	Ultrasonic Character
431	5.869	2.830	8.39	5.65	84.7	Clear
455	6.032	2.832	8.66	5.59	83.8	"
456	6.022	2.833	8.61	5.53	82.8	"
457	6.022	2.833	8.61	5.56	83.4	"
458	5.655	2.826	8.10	5.59	83.8	Substandard indications
459	5.620	2.830	7.99	5.60	83.9	Clear
460	5.620	2.820	7.99	5.58	83.7	"
481	5.982	2.835	8.55	5.49	82.1	"
482	5.910	2.830	8.42	5.57	83.5	"
483	5.950	2.828	8.48	5.54	82.9	"
484	5.830	2.826	8.31	5.54	82.9	"
485	5.855	2.830	8.29	5.51	84.0	"
486	5.943	2.833	8.41	5.52	84.2	1 Substandard indication
487	5.917	2.815	8.44	5.68	85.0	Indication exceeding std.
488	5.898	2.830	8.43	5.70	85.4	No back reflection
489	5.947	2.828	8.55	5.79	86.6	Axial spotty
490	5.967	2.831	8.53	5.65	84.6	Axial spotty

Table 17. Chrome-30 Extrusion Billet Histories - Continued

Billet Number	Billet Length, Inches	Billet Diameter, Inches	Machined Weight, Pounds	Sintered Density, g/cc	Density, As Percent of Theoretical	Ultrasonic Character
491	5.853	2.831	8.38	5.72	85.6	1 Substandard indication
492	5.980	2.825	8.55	5.72	85.6	Spotty
493	5.930	2.828	8.54	5.78	86.5	Clear
494	5.990	2.827	8.63	5.78	86.5	"
495	6.000	2.826	8.64	5.54	84.4	Small area of partial loss of back reflection
506	5.632	2.833	8.30	6.39	95.5	Substandard indication
508	5.596	2.833	8.34	6.43	96.2	
502	5.730	2.830	8.23	5.54	82.9	Axial-1 spot of signal loss
503	6.010	2.828	8.65	5.53	82.7	Clear
504	5.940	2.827	8.61	5.64	84.4	"
505	5.525	2.828	7.88	5.53	82.4	"
532	5.770	2.829	8.02	5.45	81.5	"
533	5.890	2.825	8.18	5.45	81.5	"
534	6.160	2.829	8.55	5.43	81.2	"
535	6.110	2.829	8.55	5.46	81.6	"
537	6.325	2.828	8.93	5.59	83.8	"
538	6.265	2.827	9.92	5.55	83.0	"
539	6.300	2.827	10.09	5.54	82.9	1 Indication

Table 18. Extrusion Histories of Chrome-30 Billets

ASD Extrusion Number	Billet Number	Extrusion Temperature, F	Extrusion Ratio	Ram Speed, Turns	Extrusion Pressure, Tons Maximum	Extrusion Pressure, Tons Minimum	Die Condition Before	Die Condition After
764	432	2200	10:1	1½	500	450	New	
765	437	2000	9.6:1	2	550	460	New	Good
766	438	2200	9.6:1	1	440	350	Used Once	Good
767	439	2000	9.6:1	1	530	440	Used Twice	Good
768	440	2400	9.6:1	1	390	320	Used 3 Times	Good
769	441	2400	10:1	1	400	340	Used 4 Times	Good
780	433	2000	12:1	1½	540	480	New	Slight Wash
781	434	2000	12:1	1½	510	420	Used Once	
782	443	2000	8:1	1½	480	425	New	Good
783	447	2000	8:1	1½		425	Used Once	Good
784	436	2200	8:1	1½	455	320	Used Twice	Good
785	445	2200	8:1	1½	440		Used 3 Times	Fair
786	446	2400	8:1	1½	440	300	Used 4 Times	Fair
787	454	2400	8:1	1½	440	300	Used 5 Times	Washed
808	442	2400	12:1	1½	340	280	New	Good
809	444	2400	12:1	1½	390	310	New	Good
810	430	2300	12:1	1½	390	310	Used Once	Good
811	431	2200	12:1	1½	435	390	Used Once	Good
829	455	2000	10:1	2	580	550	New	Some Mat'l Left in Die
830	456	2000	9:1	2½	590	540	Used Once	Washed
831	457	2000	9:1	2	620	540	Used Once	Good
832	458	2000	9:1	2	580	540	Used Twice	Good
833	459	2000	9:1	2	590	540	Used 3 Times	Good
834	460	2000	9:1	2	590	530	Used 4 Times	Good
844	481	2000	9:1	2	590	530	Used 5 Times	Good, Some Wash
845	482	2000	9:1	2	600	540	Used 6 Times	

Table 18. Extrusion Histories of Chrome-30 Billets - Continued

ASD Extrusion Number	Billet Number	Extrusion Temperature, F	Extrusion Ratio	Ram Speed, Turns	Extrusion Pressure, Tons: Maximum	Extrusion Pressure, Tons: Minimum	Die Condition: Before	Die Condition: After
846	483	2000	8.8:1	2	580	525	Used 3 Times	Good
847	484	2000	8.8:1	2	580	530	Used 4 Times	Good
849	485	2000	10:1	2	600	520	New	Cracked
850	486	2000	10:1	2	600	590	New	Part of Billet Stuck
851	487	2000	9:1	2	600	540	New	Good
852	488	2000	9:1	2	600	560	Used Once	Good
853	489	2000	9:1	2	620	580	Used Twice	Good
854	490	2000	9:1	2	610	570	Used 3 Times	Washed
855	491	2000	9:1	2	610	530	New	Good
856	492	2000	9:1	2	620	560	Used Once	Good
857	493	2000	9:1	2	610	540	Used Twice	Good
858	494	2000	9:1	2	590	530	Used 3 Times	Good
859	495	2000	9:1	2	590	570	Used 4 Times	Good
861	506	2000	9:1	2	600	520	Used 6 Times	Good
863	508	2000	10:1	2	610	570	New	Billet Stuck
887	502	2000	8:1	$2\frac{1}{2}$	560	495	New	Good
888	503	2000	8:1	$2\frac{1}{2}$	560	480	Used Once	Good
889	504	2000	8:1	$2\frac{1}{2}$	570	490	Used Twice	Good
890	505	2000	8:1	$2\frac{1}{2}$	545	440	Used 3 Times	Good
902	532	2000	9.4:1	2	510	400	New	Good
903	533	2000	9.4:1	2	420	380	Used Once	Good
904	534	2000	9.4:1	2	500	430	Used Twice	Fair
905	535	2000	9.4:1	2	540	460	Used 3 Times	Fair
906	537	2000	9.4:1	2	580	460	Used 4 Times	Fair
907	538	2000	9.4:1	2	510	400	Used 5 Times	Fair
908	539	2000	9.4:1	2	560	500	Used 6 Times	Fair

Table 19. Properties of Sheet Bar Extrusions

ASD Extrusion Number	Billet Number	Extrusion Density, g/cm^3	Yield, Percent	Hardness Rockwell B	Ultimate Tensile Strength, 1000 PSI	0.2% Offset Yield Strength, 1000 PSI	Elongation in 1 Inch, Percent
829	455	6.56	82.0	79.4	51,650	32,300	19.6
830	456	6.55	83.4	79.8	50,500	31,700	19.0
831	457	6.57	84.1	80.5	50,500	33,900	20.6
832	458	6.56	84.1	80.4	49,000	30,500	20.2
833	459	6.56	83.0	80.7	50,000	32,000	20.9
834	460	6.58	82.3	79.8	48,500	30,900	21.0
844	481	6.47	86.2	79.8	46,800	29,800	19.7
845	482	6.49	84.7	80.1	47,000	29,200	20.8
846	483	6.49	85.3	79.5	48,100	29,650	19.6
847	484	6.55	84.8	80.7	49,600	34,800	17.3
849	485	6.54	84.0	77.5	48,700	32,000	21.5
850	486	6.55	81.8	80.6	49,400	28,900	24.2
851	487	6.55	84.5	80.9	49,000	30,200	20.9
852	488	6.54	83.9	81.5	49,300	28,600	22.9
853	489	6.53	83.2	81.5	50,200	32,100	19.6
854	490	6.54	86.1	81.3	49,650	29,400	19.6
855	491	6.56	86.8	80.7	48,750	30,250	19.6

Table 19. Properties of Sheet Bar Extrusions - Continued

ASD Extrusion Number	Billet Number	Extrusion Density, g/cm^3	Yield, Percent	Hardness Rockwell B	Ultimate Tensile Strength, 1000 PSI	0.2% Offset Yield Strength, 1000 PSI	Elongation in 1 Inch, Percent
856	492	6.54	85.9	81.2	49,600	30,000	20.9
857	493	6.53	86.6	80.0	49,000	28,100	19.6
858	494	6.52	87.8	78.6	48,600	25,850	17.3
859	495	6.57	87.6	80.3	50,500	34,000	19.6
861	506	6.54	88.5	78.3	47,000	27,200	20.0
863	508	6.56	85.0	79.7	46,200	30,850	21.8
887	502	6.53	85.0	76.4	49,100	26,000	24.5
888	503	6.52	85.4	77.2	48,200	26,190	18.65
889	504	6.53	86.4	77.8	49,300	28,100	21.2
890	505	6.52	87.3	76.0	48,600	27,450	24.5
902	532	6.40	85.1	80.0	43,755	25,850	3.05
903	533	5.54	87.1	78.2	48,600	25,500	22.35
904	534	6.48	87.1	78.2	48,450	25,900	24.2
906	537	6.57	88.4	80.0	-	-	-
907	538	6.40	90.5	73.0	-	-	-

Table 20. Summary of Warm Rolling Breakdown Trials[a]

Trial Number	Sheet Bar Extrusion Number	Annealing Temperature[b], F	No. of Passes Before Anneal	Average Reduction Per Pass, Percent	Reduction Before Anneal, Percent	Reduction When Cracks Appeared, Percent	Final Thickness, Inches
13	832-1	1400	4	14.5	50.8	41.5	0.182
		Argon	1	12.9	12.9	12.0	0.1585
14	832-2	1800	4	15.0	47.5	47.5	0.185
		Argon	4	13.4	43.8	43.8	0.104
			2	12.9	24.0	24.0	0.079
			2	14.5	27.4	19.6	0.0575
15	832-3	1800	4	14.7	47.0	47.0	0.196
		Hydrogen	2	15.2	31.0	31.0	0.141
			1	14.0	14.9	14.9	0.120
16[c]	832-4	1800	3	16.7	42.3	42.3	0.214
		Argon					
20[c]	832-5	2000	3	10.1	27.0	27.0	0.335
		Argon	1	9.6	9.6	9.6	0.303
17	833-1	1800	4	16.1	50.4	31.2	0.238
		Argon	3	14.4	37.4	28.6	0.149
19	833-3	1800	3	15.7	40.3	40.3	0.279
		Argon	3	11.6	30.8	21.5	0.193
			2	13.3	24.8	24.8	0.145
21	834-3	1800	5	15.2	56.2	17.3	0.203
		Argon	3	13.6	35.4	26.6	0.131

Table 20. Summary of Warm Rolling Breakdown Trials [a] - Continued

Trial Number	Sheet Bar Extrusion Number	Annealing Temperature[b], F	No. of Passes Before Anneal	Average Reduction Per Pass, Percent	Reduction Before Anneal, Percent	Reduction When Cracks Appeared, Percent	Final Thickness, Inches
23	833-2	1800	3	15.5	40.5	-	0.282
		Argon	2	11.9	22.3	-	0.219
			2	13.2	24.6	-	0.165
27	829-6	1800	3	15.5	39.7	29.6	0.279
		Air	2	10.9	20.6	20.6	0.204
			2	14.0	25.8	14.7	0.151
24[c]	831-5	None	2	11.0	20.6	12.3	0.373
25	834-1	2000	3	16.2	41.5	30.4	0.275
		Argon	2	15.8	29.0	17.8	0.192
			2	15.6	27.6	27.6	0.139
			2	16.0	29.5	17.3	0.098
26	829-5	2000	3	14.9	38.4	14.7	0.268
		Argon	3	14.9	38.4	17.5	0.165
			2	16.5	30.3	17.6	0.115
			1	20.9	20.9	20.9	0.091

(a) All bars rolled at 800°F except #26 which was rolled at 1200°F.

(b) One half hour at temperature in the indicated furnace atmosphere.

(c) Rolled parallel to extrusion direction. All others rolled transverse to extrusion direction.

Table 21. Summary of Hot Rolling Trials

Trial Number	Sheet Bar Extrusion Number	Rolling Temperature, F	Number of Passes	Average Reduction Per Pass, Percent	Total Reduction, Percent	Final Annealing Temperature,(a) F	Final Sheet Thickness, Inches	Remarks
22	834-2	2200 Argon	4	20.9	61.0	None	0.181	No clad. Deep surface and edge splits.
28	829-7	2200 Argon	7	18.1	70.1	None	0.107	Nickel clad failed. Cross rolled last 38.7%.
29	831-6	2200 Argon	6	18.4	70.6	None	0.148	Nickel clad loosened. Severe surface and edge splits.
30	831-4	2200 Air	6	19.5	73.0	None	0.136	Nickel clad loosened and split. Severe surface & edge splits.
32	846-3	2200 Argon	9	21.0	87.0	None	0.060	Diffused nickel plating. Surface badly wrinkled.
33	833-4	2200 Hydrogen	9	20.4	86.3	None	0.061	Diffused nickel plating. Surface badly wrinkled.
34	863-1	2200 Hydrogen	10	17.6	86.0	None	0.061	Diffused nickel plating. Surface badly wrinkled.
35	863-3	2200 Hydrogen	10	18.6	88.8	None	0.058	Diffused nickel plating plus 40 mil sprayed nickel. Surface wrinkled.
36A	863-2	2100 Salt	2	18.1	33.0	None	0.309	Deep edge splits. Rolled parallel to extrusion.
36B	863-2	2100 alt	8	16.7	76.9	None	0.106	Deep edge splits. Rolled transverse to extrusion.
36C	863-2	2100 Salt	2	19.0	34.4	None	0.302	Deep edge splits. Rolled parallel to extrusion.
37	863-4	2200 Hydrogen	4	20.2	59.8	None	0.180	Nickel clad failed. Deep edge splits.
			The Following Trials Were Made With Sheet Bars Enclosed in Steel Frames					
38	830-1	1800	11	14.4	82.0	None	0.073	Cracked after frame removal.
41	830-2	1800	8	19.2	82.3	1800	0.066	Cracked after frame removal.
81	844-1	1900	7	19.6	77.5	None	0.077	Flattened after last pass.

Table 21. Summary of Hot Rolling Trials - Continued

Trial Number	Sheet Bar Extrusion Number	Rolling Temperature, F	Number of Passes	Average Reduction Per Pass, Percent	Total Reduction, Percent	Final Annealing Temperature,(a) F	Final Sheet Thickness, Inches	Remarks
77	863-5	1900	7	19.6	78.5	1900	0.078	Flattened after last pass.
79	887-5	1900	7	19.5	78.0	2200	0.078	Flattened after last pass.
39	850-2	2000	10	16.6	83.3	2000	0.066	Frame split.
40	850-3	2000	8	19.4	82.5	2000	0.066	Frame split.
46	849-5	2000	6	25.6	82.2	2000	0.066	Frame split.
54	844-3	2000	5	22.6	72.5	2000	0.098	
51	845-6	2000	7	24.9	86.6	2000	0.050	Cross rolled last 43%.
43	850-5	2200	11	15.4	84.0	2200	0.059	Frame split.
31	830-3	2200	7	21.1	81.0	None	0.072	
42	850-4	2200	8	19.7	82.6	2200	0.061	Frame split.
47	850-6	2200	6	25.0	82.0	2200	0.065	Frame split.
44	849-4	2200	5	29.3	82.4	2200	0.065	Frame split.
75	851-5	2200	7	19.4	77.9	2200	0.079	Flattened after last pass.
55	844-6	2200	5	23.0	73.3	2200	0.097	
56	846-5	2200	4	27.3	72.4	2200	0.100	
57	864-4	2200	4	27.2	72.0	2200	0.099	
52	849-3	2200	8	22.9	84.6	2200	0.055	Cross rolled last 52.6%
49	844-4	2300	6	28.4	86.6	2200	0.048	Frame split.
50	845-4	2300	4	38.5	85.7	2200	0.055	Frame split.
45	844-5	2300	3	35.8	73.8	2300	0.098	
53	845-5	2300	7	24.6	86.4	2200	0.049	Cross rolled last 55.7%

Table 21. Summary of Hot Rolling Trials - Continued

Trial Number	Sheet Bar Extrusion Number	Rolling Temperature, F	Number of Passes	Average Reduction Per Pass, Percent	Total Reduction, Percent	Final Annealing Temperature,(a) F	Final Sheet Thickness, Inches	Remarks
70	847-5	2200	3	25.1	58.0	-	-	
		1800	3	20.3	49.2	2000	0.077	Flattened after last pass.
71	849-1	2200	3	24.9	57.5	-	-	
		1800	3	19.7	48.2	2000	0.077	Flattened after last pass.
73	849-6	2200	3	24.8	57.5	-	-	
		1800	3	19.5	47.9	2200	0.077	Flattened after last pass.

(a) Annealed in the frame for one-half hour at temperature after the last roll pass.

Table 22. Summary of Hot-Warm Rolling Trials

Rolling Trial Number	Rolling Temperature, F	Annealing Temperature,[a] F	Number of Passes	Average Reduction Per Pass, Percent	Total Reduction, Percent	Final Thickness, Inches
Hot 78	1900	1900	7	19.5	78.0	.078
Warm 85	900	-	7	6.6	40.3	.043
Hot 80	1900	2200	7	19.6	78.0	.076
Warm 86	900	-	7	7.1	40.4	.042
Hot 72	2200/1800	2000	6	22.2	77.9	.077
Warm 82A	900	-	7	7.3	41.4	.043
Hot 74	2200/1800	2200	6	21.8	77.1	.079
Warm 83	900	-	7	6.9	39.5	.042
Hot 76	2200	2000	7	19.3	77.8	.078
Warm 84	900	-	6	7.1	38.4	.045
Hot 58	2200	2200[b]	4	27.2	72.0	-
Warm 64	900	-	10	6.5	49.0	.052
Hot 59	2200	2200[b]	4	27.0	71.8	-
Warm 65	900	-	8	7.5	46.4	.051
Hot 60	2200	2200[c]	4	27.2	72.0	-
Warm 66	900	-	5	9.4	39.0	.057
Hot 61	2200	2200[c]	4	27.3	71.8	-
Warm 67	900	-	5	7.6	32.6	.064
Hot 62	2200	2200[c]	4	27.3	71.8	-
Warm 68	900	-	5	8.5	36.1	.062
Hot 63	2200	2200[c]	4	27.5	72.5	-
Warm 69	900[d]	-	5	9.8	23.7	.059

(a) All sheets annealed for ½ hour in the frame at the indicated temperature
(b) Sheet re-annealed for ½ hour in hydrogen at 2000°F after frame removal and pickling.
(c) Sheet re-annealed for ½ hour in vacuum at 2000°F after frame removal and pickling.
(d) Warm rolled transverse to hot roll direction. All others rolled parallel to hot roll direction.

Table 23. Summary of Warm Rolling Trials

Rolling Trial Number[a]	Rolling Temperature, F	Annealing Temperature,[b] F	Final Rolling Direction[c]	Number of Passes	Average Reduction Per Pass, %	Total Reduction, %	Sheet Size, Inches[d] Length	Width	Thickness
H 102	2200	2200	-	4	29.0	74.5	4-3/4	5-3/4	0.084
W 117	600	-	P	5	9.9	40.9	6-1/2	5-1/16	0.048
H 103	2200	2200	-	4	28.8	74.4	4-3/8	5-7/8	0.086
W 118	600	-	P	5	10.1	41.3	5-7/16	5-3/4	0.051
H 108	2200	2200	-	4	28.5	74.1	5	5-3/4	0.085
W 137	600	-	T	5	9.5	39.2	8-1/2	4-1/2	0.052
H 104	2200	2200	-	4	28.8	74.4	5	5-7/8	0.086
W 119	900	-	P	6	8.1	39.6	6-7/8	5-3/4	0.051
H 105	2200	2200	-	4	28.6	74.4	5	5-1/2	0.085
W 120	900	-	P	6	9.0	43.2	6-7/8	5-1/4	0.049
H 109	2200	2200	-	4	29.0	74.5	4-3/4	5-7/8	0.084
W 141	900	-	T	5	9.3	39.0	6-3/8	4-5/8	0.051
H 106	2200	2200	-	4	28.8	74.4	4-3/4	5-7/8	0.084
W 115	1200	-	P	5	8.9	37.2	6-3/8	5-7/8	0.0535
H 107	2200	2200	-	4	28.9	74.5	5-1/8	5-7/16	0.086
W 116	1200	-	P	5	8.9	37.2	5-1/2	4-3/4	0.0535
H 110	2200	2200	-	4	28.7	74.3	5	5-5/8	0.085
W 139	1200	-	T	5	9.5	39.6	9	4-1/2	0.0495
H 113	2200/1800	1800	-	5	24.6	74.6	5-1/2	5-7/8	0.085
W 133	900	-	T	7	7.1	40.3	7-3/4	6	0.048

Table 23. Summary of Warm Rolling Trials - Continued

Rolling Trial Number[(a)]	Rolling Temperature, F	Annealing Temperature,[(b)] F	Final Rolling Direction[(c)]	Number of Passes	Average Reduction Per Pass, %	Total Reduction, %	Sheet Size, Inches[(d)] Length	Width	Thickness
H 114	2200/1800	1800	-	5	24.6	74.7	5	5-1/2	0.084
W 134	900	-	P	6	7.9	39.3	6-1/2	4-7/8	0.050
H 93	2200	2200	-	5	27.9	80.5	6-3/8	5-1/2	0.065
W 121	600	-	T	2	8.9	17.0	4	5-1/2	0.053
H 94	2200	2200	-	5	28.1	80.7	6-1/2	5-7/8	0.065
W 122	600	-	T	2	10.4	20.0	4-11/16	4-3/4	0.0515
H 99	2200	2200	-	5	27.9	80.5	6-1/4	5-3/8	0.065
W 136	600	-	P	3	9.0	23.1	5-7/8	4-3/8	0.048
H 95	2200	2200	-	5	27.9	80.4	6-1/2	5-1/2	0.065
W 125	900	-	T	3	8.4	23.3	6-5/8	2-7/8	0.051
H 96	2200	2200	-	5	28.0	80.6	6-1/8	6	0.061
W 126	900	-	T	3	8.5	23.7	5-3/4	5-3/4	0.052
H 100	2200	2200	-	6	23.9	81.0	6-3/8	6	0.0625
W 140	900	-	P	2	10.5	20.0	6-3/8	5-7/8	0.050
H 98	2200	2200	-	5	27.9	80.8	6	5-7/8	0.065
W 124	1200	-	T	3	7.6	21.2	5-1/2	5-13/16	0.0515
H 101	2200	2200	-	6	24.6	82.1	7	5-7/8	0.0575
W 138	1200	-	P	2	9.6	18.3	7-1/2	5-13/16	0.047
H 112	2200/1800	1800	-	6	24.0	80.9	6-1/2	6	0.0625
W 132	900	-	T	5	4.9	22.4	8-1/2	6	0.048

H 89	2200	2200	-	6	25.7	83.0	7-5/8	5-1/2	0.054
W 127	900	-	T	1	10.0	10.0	4-3/4	4-1/4	0.049
H 90	2200	2200	-	6	25.4	82.8	7-3/16	5-1/4	0.056
W 128	900	-	T	1	8.9	8.9	7-1/4	5-1/4	0.050
H 91	2200	2200	-	6	26.0	83.5	6-1/2	6	0.053
W 129	1200	-	T	1	9.2	9.2	5-1/4	3-1/4	0.047
H 92	2200	2200	-	6	25.8	83.2	7-1/2	5-1/4	0.057
W 130	1200	-	T	1	9.7	9.7	5-15/16	3-3/16	0.0495
H 111	2200/1800	1800	-	6	25.5	83.1	6-3/8	5-1/2	0.066
W 131	900	-	T	1	6.9	6.9	6-1/4	5-1/2	0.046

(a) H designates Hot Rolling Trial Number; W designates Warm Rolling Trial Number.
(b) All sheets annealed for ½ hour in the frame at the indicated temperature and then re-annealed for ½ hour at 2000°F in vacuum after frame removal and pickling.
(c) P - Parallel to hot roll direction; T - Transverse to hot roll direction.
(d) Pre-trimmed dimensions reported.

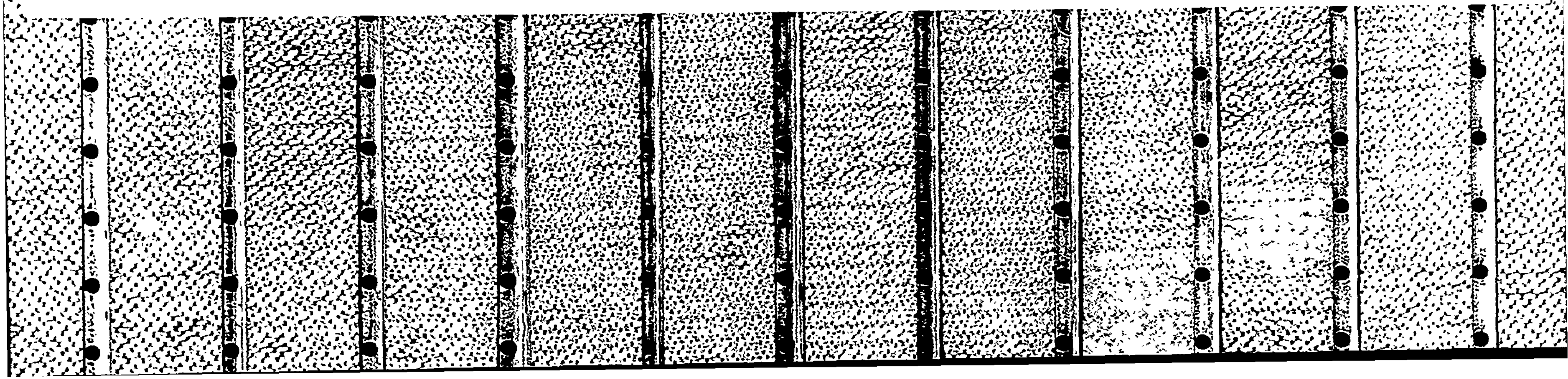


Table 24. Summary of Special and Optimum Rolling Trials

	Hot Rolling					Warm Rolling					
Sheet Bar	Sheet Number	Rolling Temp., F	No. of Passes	Average Reduction Per Pass, Percent	Total Reduction, Percent	Sheet Number	Rolling Temp., F	No. of Passes	Average Reduction Per Pass, Percent	Total Reduction, Percent	Final Thickness, Inches
Recrystallization Study											
857-6	151	2200	5	29.4	82.6	163	900	1	8.8	8.8	0.049
857-4	149	2200	5	28.2	80.9	164	900	2	9.3	17.3	0.052
859-1	142	2200	4	28.4	74.0	165	900	5	9.3	38.8	0.052
859-2	143	2200	3	26.9	63.0	166	900	9	9.9	61.0	0.061
859-4	144	2200	1	27.8	27.8	167	900	11	10.1	70.0	0.076
Heat Treatment Study											
906-3	187	2200	4	28.4	73.6	203	900	7	7.6	41.5	0.050
904-2	188	2200	4	28.6	73.9	208	900	7	6.6	41.3	0.049
907-2	190	2200	4	26.3	70.6	209	900	7	7.8	43.1	0.049
861-2	194	2200	4	26.7	71.3	210	900	7	7.3	41.3	0.049
861-4	198	2200	4	26.4	70.8	211	900	7	7.3	41.2	0.049
854-1	159	2200	4	28.5	73.9	169	900	7	6.7	38.7	0.054
Ductile-Brittle Transition Study											
906-4	193	2200	4	27.6	72.6	212	900	7	7.2	40.6	0.049
904-1	196	2200	4	26.1	70.4	213	900	7	7.3	41.4	0.049
906-5	197	2200	4	26.2	70.5	214	900	7	7.9	43.8	0.049
861-1	195	2200	4	26.5	71.0	206	900	11	6.6	52.3	0.040
858-4	153	2200	5	30.0	83.1	173	900	1	7.9	7.9	0.049
Oxidation Study											
906-6	191	2200	4	28.1	73.3	204	900	7	7.4	40.4	0.051

Table 24. Summary of Special and Optimum Rolling Trials - Continued

	Hot Rolling					Warm Rolling					
Sheet Bar	Sheet Number	Rolling Temp., F	No. of Passes	Average Reduction Per Pass, Percent	Total Reduction, Percent	Sheet Number	Rolling Temp., F	No. of Passes	Average Reduction Per Pass, Percent	Total Reduction, Percent	Final Thickness, Inches
Elevated Temperature Strength Study											
907-4	176	2200	4	28.6	73.9	200	900	7	7.0	39.8	0.051
888-2	186	2200	4	28.2	73.4	202	900	7	7.4	40.4	0.051
888-1	192	2200	4	27.6	72.6	205	900	7	7.6	41.5	0.051
902-5	199	2200	4	24.6	67.9	207	900	7	6.8	38.6	0.050
Stress Rupture Study											
829-4	175	2200	4	28.4	73.6	215	900	7	7.6	42.6	0.049
907-5	177	2200	4	28.3	73.5	216	900	7	7.9	43.9	0.049
907-6	178	2200	4	28.5	73.8	217	900	7	7.6	42.6	0.048
907-3	179	2200	4	28.5	73.8	218	900	7	7.5	41.8	0.049
906-2	180	2200	4	28.6	74.0	219	900	7	7.5	42.0	0.049
906-1	181	2200	4	28.3	73.5	220	900	7	6.8	40.2	0.049
904-5	183	2200	4	28.6	73.9	221	900	7	7.8	43.1	0.049
904-6	184	2200	4	28.3	73.7	222	900	7	7.5	41.9	0.049
904-3	185	2200	4	28.1	73.3	223	900	7	7.2	40.9	0.049
907-7	189	2200	4	28.6	73.9	224	900	7	7.5	42.0	0.049

Aeronautical Systems Division, Dir/Materials and Processes, Metals and Ceramics Laboratory, Wright-Patterson AFB, Ohio.
Rpt No. ASD-TDR-63-297. DEVELOPMENT OF CHROMIUM COMPOSITE ALLOY WITH HIGH TEMPERATURE OXIDATION AND EROSION RESISTANCE. Final report, April 63, 138pp. incl illus., tables, 2 refs.

Unclassified Report

The effects of extrusion and rolling variables on the quality and mechanical behavior of a powder metallurgy chromium-magnesium oxide composite have been studied.

Hot rolling at 2200 F and finish rolling at 900 F with reductions of 40 to 55 percent provided sound, contamination free sheet having a ductile-brittle transition temperature of 45 F in the recrystallized

(over)

1. Chromium Alloys
2. Oxidation-Reduction Reactions
3. Erosion
4. High Temperature Research
I. AFSC Project 7381, Task 738102
II. Contract AF33(657)-8422
III. Bendix Products Aerospace Division, The Bendix Corp, South Bend, Indiana
IV. James F. Masterson
V. Aval fr OTS
VI. In ASTIA collection

condition. Oxidation, erosion and nitridation behavior were observed to be improved over unalloyed chromium. Preliminary studies have indicated that a strain aging phenomenon may be responsible for the brittle behavior observed with as rolled and stress relieved sheets. Further work is required to resolve this anomaly.

The results of this initial program have indicated that the full potential of chromium composites can be realized with additional development directed toward strengthening, and further retardation of nitrogen diffusion at elevated temperature.

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